COMPUTER AIDED INDEXING OF LAUE BACK REFLECTION PATTERNS FOR A BODY CENTERED TETRAGONAL CRYSTAL

by

P. SAMPATH KUMAR

DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

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by

P. SAMPATH KUMAR

to the

DEPARTMENT OF METALLURGICAL ENGINEERING
INDIAN INSTITUTE OF TECHNOLOGY, KANPUR

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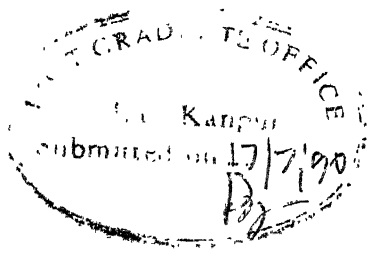
To

My Sisters,

Sujatha and Padmaja

and their lovely kids.



CERTIFICATE

It is certified that the work contained in the thesis entitled "Computer aided indexing of Laue back reflection patterns for a body centered tetragonal crystal", by Mr. P.Sampath Kumar has been carried out under my supervision and that this work has not been submitted elsewhere for a degree.

Dr. V.Bansal

Department of Metallurgical Engineering

Indian Institute of Technology

Kanpur.

July, 1990.

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CHAPTER 1 : INTRODUCTION

Determining the crystal orientation by the Laue method involves indexing some of the Spots and/or some of the Zones of the Spots on the Laue photograph. Indexing a crystal which is oriented at random with respect to the X-Ray beam proves difficult. The routine for indexing Laue back reflection patterns can be tedious and time consuming. Even for a skilled operator, conversant with Greninger chart, Wulff net and Stereographic projections, it often requires considerable time and experience before difficult patterns can be solved. Indexing of Laue patterns for cubic crystals is generally easier, since the tables of interplanar angles and standard stereographic projections are available. When the crystal structure is non-cubic, tables of interplanar angles may not be available and indexing a pattern is then not possible. Since the labour involved in indexing Laue patterns is enormous, various approches have been made to index the pattern by computer.

The present work involves developing a computer algorithm for the rapid solution of Laue back reflection patterns of body centered tetragonal crystals.

Reflections of planes having indices (6 6 6) to ($\bar{6}$ $\bar{6}$ $\bar{6}$) are indexed directly by using the X and Y coordinates of the Spots on the film as the input data. The code developed finds out the indices of the important zone axes and the mutual angles of these axes are also matched with the given angles obtained from the stereographic projection of the pattern. The computational scheme compares the interplanar angles for the various spots on the film with the theoretical interplanar angles, which are calculated by the program after assigning Miller indices to the planes causing these spots. The systematic error associated with measurement of the distance of seperation between the sample and the X-Ray film is minimised to determine the best value of distance.

Directionally solidified alloys of Tin have dendritic structure. The primary dendrites are plate like in shape and are roughly parallel to the growth direction, which in turn is the ingot axis. It is noted from the literature that, the growth direction in these alloys is [1 1 Ø] This value has been reported for pure Tin [1], as well as for Sn-12% Bi alloys [2,3]. The present work involves developing the computer technique for indexing the LBR patterns of these alloys and also to check the obtained crystallographic

orientation with the published data.

Tin alloys with 2%, 6% and 12% Bismuth were used for obtaining the Laue back reflection patterns for subsequent indexing. These alloys are directionally solidified at different freezing rates. The crystallographic orientation of each sample is determined with the help the indices of the zone axes obtained from the program. This is then compared with the published data for the orientation of Sn-Bi alloys.

The computer program entitled " *LAUE* ", is written in FORTRAN 77 and the executions are carried out in Hewelett Packard 9000 system. The execution takes about 2 to 3 seconds of CPU time for indexing 7 spots, with an allowable error of 3.0 degrees.

CHAPTER 2 : LITERATURE SURVEY

The various methods of indexing Laue patterns using computer are briefly reviewed in this chapter.

A number of workers have tried indexing the Laue patterns by computer. Many of the techniques adopted are for a specific crystal system and the others are for any crystal system. One of the early attempts is by D.T.Camp and J.A.Clum [4]. They have developed a scheme for indexing LBR patterns of Zinc crystal (hexagonal). The scheme first calculates the interplanar angles between the planes with indices, within a specified limit (3 3 3) to ($\bar{3}\ \bar{3}\ \bar{3}$) and stores them. These angles are then compared with the experimental interplanar angles measured from a stereographic projection. The measured input data is systematically compared with the generated table of standard angles. All combinations of poles whose interplanar angles compare with the requisite specified accuracy, are printed out. The computer program was written in FORTRAN IV.

Christensen et al. [5,6] have developed a technique

which duplicates the traditional technique of matching the angles between prominent reflections on the film, with the true values of interplanar angles. The scheme seeks a solution by assigning trial indices to a pair of reflections generating the theoretical interplanar angle and comparing it with its counterpart from the X-Ray pattern. They have limi.ed their search between (3 3 3) to ($\bar{3}$ $\bar{3}$ $\bar{3}$), for the planes causing the spots. They have adopted this scheme for indexing Silver single crystals (cubic). The same technique has been used by Cyril Anazia et al. [7] for indexing Sapphire single crystals (hexagonal). The computer program was written in FORTRAN IV.

Haskell V. Hart et al. [8] have developed a scheme of indexing LBR patterns of echinoid Calcite (cubic). Here they identify the spots belonging to each zone and they assign indices to these zone axes. The angles between zone axes are measured and compared with the experimentally calculated angles, (using the γ and δ values of the hyperbolas from the Greninger's chart). They have written the computer program in BASIC. A similar approch of indexing the zone axes has been adopted by Fewster [9] also.

Jean Laugier et al. [1Ø] have developed a scheme

which does the indexing of LBR patterns of any crystal system. The computer program was written in FORTRAN IV. The data observed are (X,Y) co-ordinates of the spots on the Laue photograph. Two reflections which are at the intersection of zonal planes and thus, having small Miller indices are indexed first. The angle between the corresponding scattering vectors is compared, within a given angular tolerance, to the angles between all possible pairs of reciprocal vectors upto a given limit for the Miller indices. The verification of calculated solutions is made by means of the visual comparison between the experimental Laue photograph and the simulated patterns displayed on the same scale. Thus, by simple super imposition of the observed and the calculated patterns, all the observed spots are checked visually.

The computer program developed by C.A. Cornelius [11] also involves comparison of the Laue pattern generated by computer with the experimentally obtained photographs. This scheme is for indexing LBR patterns of any crystal structure.

The present work incorporates the scheme similar to the one developed by Christensen et al. [5,6], which requires

measuring the X and Y co-ordinates of the spots on the film. In addition to matching the interplanar angles, the zone axes are also indexed and the mutual angles between these axes are matched with the values obtained from the stereographic projection of the pattern.

The present work also involves determining the crystallographic orientation of Sn-Bi alloys and comparing it with published data. The primary dendrites of directionally solidified alloys of Sn are plate like in shape and are roughly parallel to the growth direction. G.F. Bolling et al. [1] have determined the growth direction of pure Tin (99.999 + purity) to be [1 1 Ø]. P.J. Ahearn et al. [2] have confirmed this growth direction for Sn-12% Bi alloy also. J.C. Warner et al. [3] have also reported [1 1 Ø] as the growth direction for Tin rich alloys.

The computational scheme involves calculation of theoretical interplanar and interzonal angles. These values have been checked with published values for Tin crystals by R.E. Trounfelker et al. [13] and B.S. Chandrasekar et al. [14]. The lattice constants c and a are obtained from the work published by J.A. Lee et al. [15]. Lee et al. report a change in c/a ratio from Ø.5455 for pure tin to Ø.5451 for 6% Bi in Tin. The effect of such a variation in c/a ratio on an

interplanar angle of 28.8298 degrees (between (1 2 3) and (3 3 2)) is found to be Ø.ØØ742 degrees. Since this variation in interplanar angle due to a change in c/a ratio is negligible compared to the permissible error of 3.Ø degrees used in the computer code, it was decided to use c/a ratio corresponding to pure Tin itself, which is Ø.5455.

CHAPTER 3: EXPRIMENTAL WORK

The experimental work carried out in this project involves,

(i) preperation of the sample for X-Ray exposure,

(ii) obtaining the Laue back reflection pattern from the sample,

(iii) obtaining the input data from these LBR patterns.

3.1 Preperation of the samples :

Directionally solidified Tin - Bismuth samples were used for taking the Laue back reflection patterns. Directionally solidified ingots of Tin with 2%, 6% and 12% Bismuth had been prepared by Mr. N.Das in the Solidification Laborotory earlier. The ingots were solidified at different freezing rates. TABLE I shows the freezing rate for different compositions.

TABLE 1. EXPERIMENTAL SAMPLES

Wt % Bi	Rate of freezing (μ/s)	Sample No.	Computer output No.(Appendix III)
2	41.7	4.3	5
	68.4	2.1	3
	255.Ø	3.1	4
6	41.7	6.4	1Ø
	41.7	6.4 M	8
	68.4	6.2	6
	255.Ø	6.3Ø	7
12	41.7	12.2	-
	68.4	12.33	-
	255.Ø	12.4	9

These ingots were about 1 cm in diameter and 15 cm in length. The ingots were cut into small samples of approximately 2.5 cm in length. The ingots were cut into two halves along its axis and one half of it is used. Figure.1 shows a typical sample size.

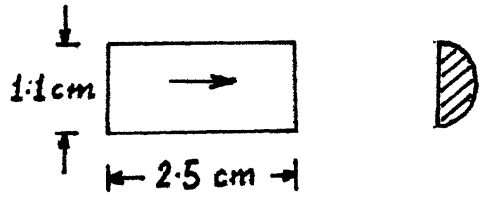


Figure. 1.

Each sample is marked with an arrow which determines the face which, is to be polished for X - Ray exposure.

The preparation of the sample for X-Ray exposure involves polishing and etching of the transverse surface (cross hatched in Fig. 1). Since the material is very soft, care has to be exercised at each stage of polishing. The initially cut sample is polishied with emery cloth. Then polishing is carried out in 1/Ø , 2/Ø and 3/Ø emery papers. After fine emery polishing, the samples are polished in wheel polishers, fitted with a silk cloth, using Alumina as the polishing medium. This gives a fairly good, scratch free surface.

The samples are etched and polished alternatively. At each stage, the microstructure is observed at a low magnification (5Ø - 1ØØX). Two different etchants were used, one for 2% and 6% Bi aaloys and the other for 12% Bi alloy. The etchants used are as follows :

% Bi	Etchant
2 and 6%	$K_2Cr_2O_7$ 1 gm + Conc. HCl 6-7 ml + Distilled water 86 ml.

12%	$FeCl_3.6H_2O$	2 gm
	Conc. HCl	5 cc
	Distl. water	3Ø cc
	C_2H_5OH (95%)	6Ø cc
	(Taffs Reagent)	

The microstructure of the polished surface is observed at 5Ø - 1ØØ X and the largest grain is located. Whithin this grain, the suitable spot for X-Ray exposure is located. For most of the samples, the grain at the geometric centre is selected, since alligning the X-Ray beam to fall on the geometric centre is much simpler. If a suitable grain is not available at the geometric centre, then an alternative grain is selected and its position is noted, by drawing the microstructure of the surface and measuring the approximate location of the spot.

3.2 Obtaining the Laue back reflection patterns :

The Laue back reflection film cassette has the dimension for a film of 8.3 X 1Ø.1 cm. The X-Ray film is cut as closely to this size as possible and a centre hole is punched. The X-Ray film is enclosed in a black paper envelope

to prevent blackening due to light leaking through the sides of the cassette. A black paper is kept at the centre also, with a hole of the size of the collimeter to prevent any leakage of light.

The samples were exposed to X-Rays at 2Ø mA and 3Ø kV for a period of 7 to 8 hours. Cu target X-Ray tube is used. The intensity of the spots obtained is observed to be low and hence the current and voltage were increased to 3Ø mA and 4Ø kV for some of the samples. THe exposure time was also increased to 9 hours.

3.3 Obtaining the input data :

The input data are obtained from the LBR pattern by,

(i) tracing the locations of the spots on a tracing paper to measure the X and Y coordinates of each spot,

(ii) preparing a stereographic projection, to locate the zone axes and to measure the angles between them.

The X-Ray film is kept over a glass plate and a tracing papar is fixed over the film. By illuminating the plate from the bottom, the position of each spot is traced on

the paper. The X and Y axes of the film (assumed to be parallel to the edges of the film) are also drawn on the paper. The origin is plotted at the centre of the hole in the film. Thus a replica of the film is prepared.

This replica is then superimposed over a graph paper with 1.Ø mm division. The origin in the pattern is coincided with the origin on the graph paper. Then the X and Y coordinates of each spot is measuredwith an accuracy of $\pm$ Ø.5 mm using eye ball judgement. Appendix I shows the transparencies prepared for each film, with the X and Y coordinates of the spots.

By using a Greninger's chart, the γ and δ values of the spots are measured. These values are then transferred on a stereographic projection. THe spots belonging to one great circle are located and all possible great circles are traced, with each great circle containing atleast three spots. The great circles containing more than 4 or 5 spots are considered as low index zones. The spots lying at the intersection of more than 2 zones are considered as the prominent spots. The zone axis for each zone is located on the stereographic projection and the mutual angle between

these axes are measured.

The proper selection of the desired number of spots is important for the indexing of the spots. The spots are numbered arbitrarily in the beginning. The spots lying at the intersection of two or three zones are renumbered first and the rest of the spots are numbered in that sequence. A minimum of six spots are selected such that they involve atleast two to three zones. The zone axes are labelled with alphabets.

CHAPTER 4 : COMPUTER PROGRAM

4.1 Technique adopted :

An iterative scheme of matching the interplanar angles between the planes causing the spots, obtained,

(i) using their X and Y co-ordinates on the X-Ray film and

(ii) with the assigned Miller indices, has been used in the Computer code developed. The scheme measures the angle between the spots using their X and Y co-ordinates, which is designated as ϕ_E , representing Experimental angles. Trial indices are assigned to the spots in a sequence and using these indices the mutual angles are measured using the crystallographic expression for the tetragonal crystal (eqn. 8). These angles are designated as ϕ_T, representing Theoretical angles. These two angles are compared such that they are within an allowable error limit ($\pm$ 3.Ø degrees). This scheme is carried out for all the spots.

In addition to matching the interplanar angles between the spots, the conditions that, certain defined spots should belong to the same zone and the mutual angle of their zone axes should match with the given values are also carried out.

For each Laue film, a stereographic projection is prepared. From this the spots belonging to the same zone are identified by tracing the spots falling on one great circle. The zone axes are also located, on the stereographic projection. Then the mutual angle between these zone angles are measured on the stereographic projection, which in turn must match the ones calculated theoretically, using the appropriate expression in the program.

The scheme also incorporates optimisation of the film to sample distance. This is done by minimising the sum of squared errors between the experimental angles (ϕ_E) and the theoretical angles (ϕ_T). The indices of each zone axis are also calculated and their mutual angles are calculated.

This chapter explains the method of measuring the angles using the X and Y co-ordinates, selection of indices, indexing scheme,determining the crystallographic orientation, and adjustment of film to sample distance.

4.1.1 Measurement of mutual angles :

(i) Experimental angles :

The experimental angles refer to the mutual angles between the spots obtained from their X and Y co-ordinates. The method adopted is the one developed by Christensen et al.

[5,6]. The computation of angles associated with pairs of reflectionsis accomplished in a perfectly straight forward manner. The geometry involved in the calculation of the angles is explained in Figure 2. The film plane is assumed to have the X and Y axes. The beam direction is taken as the Z axis. The specimen to film distance is given as D. Each spot falls on the film with corresponding X and Y values. The angle $2\phi_{i,j}$ is the angle between two reflections from the sample S. These two reflections have X and Y values as (X_i Y_i) and (X_j Y_j), with respect to the origin O, which is the point through which the X-Ray beam passes through the film. From figure 2.a,

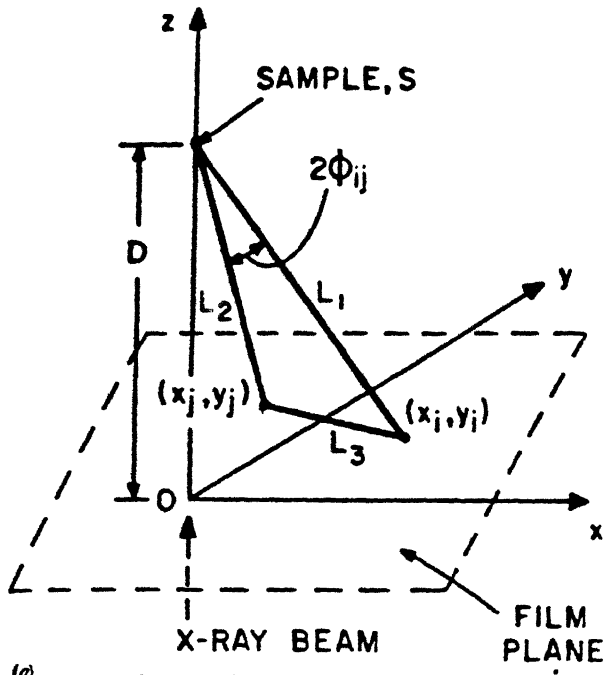
$$L_1^2 = X_i^2 + Y_i^2 + D^2 \qquad (1)$$

$$L_2^2 = X_j^2 + Y_j^2 + D^2 \qquad (2)$$

$$L_3^2 = (X_i - X_j)^2 + (Y_i - Y_j)^2 \qquad (3)$$

Figure 2.b gives the construction of the two spots' locations and the sample, in which

$$L_4 = \frac{L_1^2 - L_2^2 + L_3^2}{2L_3} \qquad (4)$$

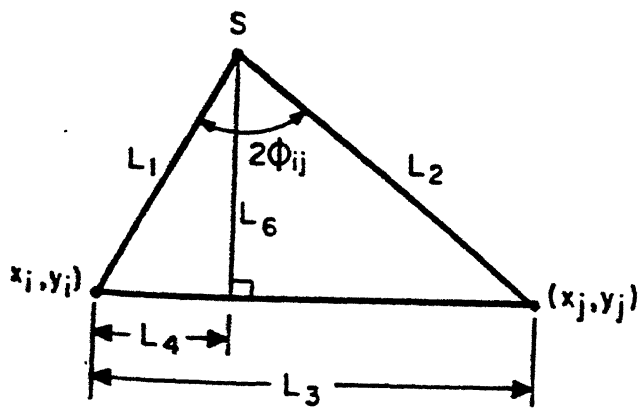


(a)

$$L_1^2 = x_i^2 + y_i^2 + D^2$$

$$L_2^2 = x_j^2 + y_j^2 + D^2$$

$$L_3^2 = (x_i - x_j)^2 + (y_i - y_j)^2$$



(b)

$$L_4 = \frac{L_1^2 - L_2^2 + L_3^2}{2L_3}$$

$$L_6^2 = L_1^2 - L_4^2$$

$$L_6^2 = L_1^2 - L_4^2 \qquad (5)$$

$$2\phi_{ij} = \tan^{-1}\left(\frac{L_4}{L_1}\right) + \tan^{-1}\left(\frac{L_3 - L_4}{L_6}\right) \qquad (6)$$

$$\phi_{ij} = 1/2\left(\tan^{-1}\left(\frac{L_4}{L_1}\right) + \tan^{-1}\left(\frac{L_3 - L_4}{L_6}\right)\right) \qquad (7)$$

ϕ_{ij} represents the angle between the spots, i and j. These angles are measured for all the spots to be indexed. These are the experimental angles ϕ_{ij}, i = 1,2, ..., N and j = i+1, i+2, ..., N, where N is the total number of spots to be indexed.

(ii) Theoretical angles :

For a set of indices assigned to two spots, the angle between them is measured using the crystallographic expression for the tetragonal crystal. These angles are designated as theoretical angles ϕ_T. The expression for measuring the interplanar angle is

$$\cos(\phi_{ij}) = \frac{(h_i h_j + k_i k_j)(c/a)^2 + (l_i l_j)}{\sqrt{[(h_i^2+k_i^2)(c/a)^2+l_i^2]\,[(h_j^2+k_j^2)(c/a)^2+l_j^2]}} \qquad (8)$$

where h,k,l are the indices assigned the spots i and j.

(iii) Zone angles :

The scheme incorporates angular match between the zone axes also. Hence for a set of indices $(u_i v_i w_i)$ and $(u_j v_j w_j)$ assigned to two zone axes, their mutual angle is given by the expression,

$$\cos(\phi_{ij}) = \frac{(u_i u_j + v_i v_j)/(c/a)^2 + w_i w_j}{\sqrt{[(u_i^2+v_i^2)/(c/a)^2 + w_i^2]\,[(u_j^2+v_j^2)/(c/a)^2 + w_j^2]}} \qquad (9)$$

4.1.2. Selection of indices :

The index search is restricted between (6 6 6) to ($\bar{6}$ $\bar{6}$ $\bar{6}$), since a judicious choice of spots on the film will normally ensure that their indices lie within this range. The total number of indices possibile in this range are 1331 including (Ø Ø Ø). For body centered crystal, from the structure factor expression, planes with (h + k + l) = odd do not produce any reflections. Hence all those indices with (h + k + l) = odd are excluded from the range of indices.

Considerable savings in search time can be achieved without any loss in generallity if the scalar multiple of

previously considered indices are excluded from the search. In general if (h k l) is considered, then (nh nk nl) need not be considered, where n is an integer. These conditions greatly reduce the number of index sets to be considered.

The selected index sets within this specified limits are arranged from lower indices to higher indices, so that the lower indices are first assigned to the spots. For a given index set, (h k l), larger the d spacing, lower will be the indices. Based on this criteria, the index sets are arranged in descending order of d spacing, which is calculated for each index set from the expression,

$$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \qquad (10)$$

where h,k,l are the indices and a and c are the lattice constants.

4.1.3. Scheme of indexing :

The scheme finds out trial indices for the N spots, such that all the experimental and theoretical angles are within an allowable arror range. The error criterion is defined as,

$$\left| \phi_T - \phi_E \right| \leq \text{ERROR} \qquad (11)$$

The allowable error range is fixed as $\pm$3.Ø degrees. All the spots are initiallised with the indices (Ø 1 1), which is the first index set to bo assigned. The heirarchy of indexing is explained in Figure 3. The scheme tests the angular match between a pair of spots at any stage. First it checks the angular match between spot numbers 2 and 1. If the angle between these spots are not matching with the their experimental angles, then the indices of spot number 2 is changed. With this index, the angle between spot 2 and 1 is again checked. If a match is not found with all the possible index sets being assigned to spot number 2, then the indices of spot number 1 is changed to the next set in the list and the processing of assigning indices to spot 2 is repeated.

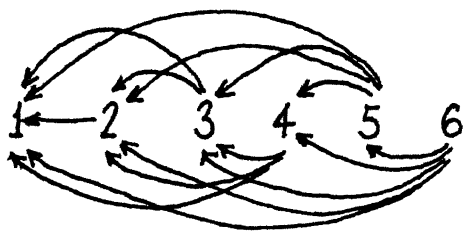


Figure 3

Angular match should be obtained between the spots in the tail end and head end of the arrows.

If a suitable match is found between spots 1 and 2, then with their indices fixed, the scheme moves to the next spot, which is spot number 3. Now the angular match between spot number 2 and 3 is checked, by assigning indices to spot number 3. With a pariticular index set assigned to spot number 3, if an angular match is obtained between spots 2 and 3, then with that index set for spot number 3, the angular match will be checked between spot number 3 and 1. If the angular match between these two spots is not acceptable, then the indices of spot number 3 will be changed and the angular match between spots 2 and 3 will be checked. The indices assigned to spot number 3 will be changed, if the angular match between spots 3 and 1 are not acceptable, even though the match between spots 3 and 2 is satisfactory.If the entire range has been exhausted for spot number 3 and if still a match is not obtained, then the indices of the spot immediately before it will be changed, ie the indices of spot number 2, and the angular match proceeds from this spot onwards. Fixing the indices to a spot implies that the indices assigned to that spot satisfies the angular match with all the previous spots. For example assigning indices to spot number 5 would mean, angular match between spots 5 and 4, 5 and 3, 5 and 2, and 5 and 1. This way the scheme proceeds the indexing of all the spots.

In addition to matching the interplanar angles, the scheme also assigns indices such that, they satisfy the zone condition, if a set of spots belong to one zone. The numbers of the spots belonging one zone are obtained from the stereogaphic projection of the pattern. For eample in set of 6 spots, if spots 1, 4, 5 and 2, 3, 6 are belonging to one zone, then the indices are assigned to the spots so that this condition is satisfied. In the indexing scheme, for eample if spots 1, 4, and 5 are belonging to one zone, then when assiging the indices to spot number 5, satisfying the angular match satisfactorily, with the indices of spot 1, 4, and 5, the condition, that they belong to the same zone is checked. This check is done by calculating the determinant of the matrix formed by the indices of the three spots. If the zone condition is not satisfied, then the indices of spot number 5 will be varied and the angular match explained in the previous paragraph will be again carried out. This way the assigned indices satisfy the zone condition also for certain specified spots.

The indices of each zone axis will also be calculated and the angle between these zone axes is matched with the given values. Essentially zone checking includes assigning the indices to the spots such that they are belonging to one zone and the angles between their zone zxes match with the

given values obtained from the stereographic projection.

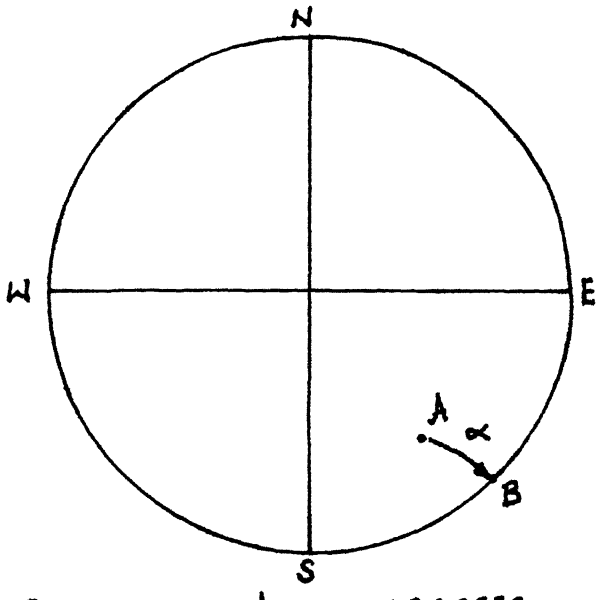
The zone checking condition is incorporated in the scheme to obtain a unique set of indices for the spots. The scheme explained, incorporates some of the features of manual indexing. The scheme fecilitates the possibility of obtaining a unique set, irrespective of the sequence in which the spots are numbered and fed. Since it is quite possible to obtain a completely different set, which also satisfies all the required conditions, when the sequence in which the spots are numbered is changed, the zone checking condition is important. It is observed, that inclusion of this condition results in a unique set of indices for the spots, irrespective of the sequence of numbering them.

The scheme explained is one of trial and error method and the proper selection ofspots for feeding into the program is very important for obtaining a set of indices, satisfying all the required conditions. The spots which lie at the intersection of two or three zones will be lablled first. The selected spots will include a minimum of 2 zones, so that from the indices of these zone zxes, the orientation of the crystal can be determined.

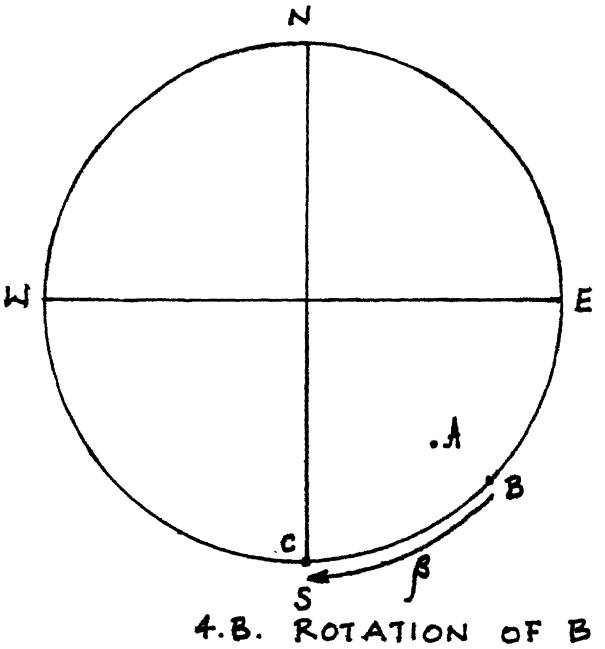
4.1.4. Determining the crystallographic orientation :

The scheme, after indexing the required number of spots

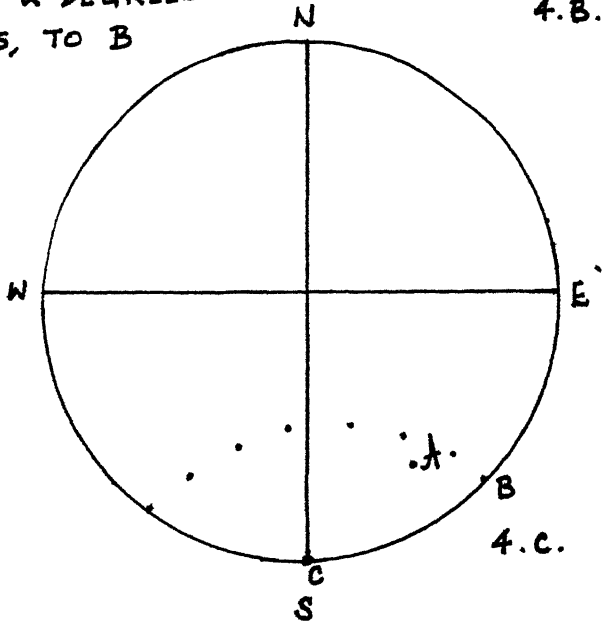
gives the indices of the zone axes also. From these indices of zone axes, the orientation can be determined easily. The samples used in the present work were Sn-Bi alloys, which have their primary dendrites roughly parallel to the growth direction, which is [1 1 Ø], as reported in the literature [1,2,3]. The angle between the indexed zone axes and [1 1 Ø] is calculated . The method of determining the orientation is as follows: the stereographic projection of the pattern is fixed on a Wulff net, such that the X and Y axes of the projection coincide with the E-W and N-S directions of the net respectively. The pole of the axes is first rotated with respect to the N-S axis to bring the pole on the curcumferance of the net. (refer to Figure 4). The angle by which the pole is rotated about the N-S axis is noted. The pole is then made to coincide on either the North or South pole of the net. With this position of the pole, the small circle which is at the required angular distance from the pole is traced. Few points are marked on this small circle. Now the pole is rotated back with respect to the axis perpendicukar to the projection. The pole is again brought to its initial position by rotating with respect to the E-W axis by the same number of degrees, by which it was rotated before. The points which are marked on the small circle are also rotated by the same number of degrees about the N-S



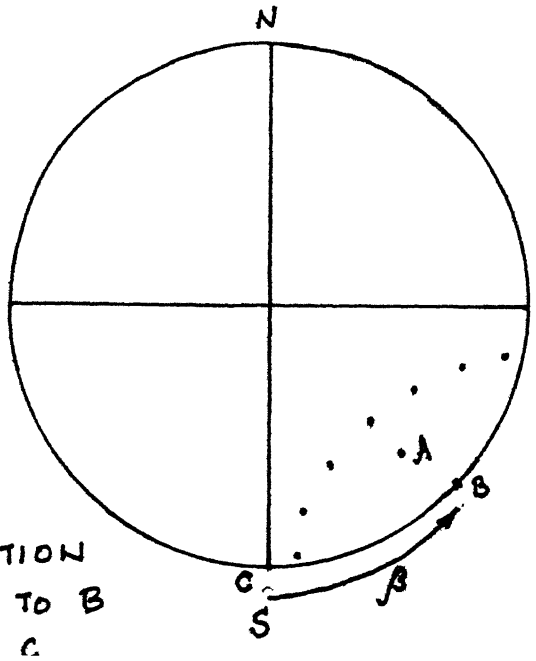
. ROTAION OF A BY α DEGREES ABOUT N-S AXIS, TO B



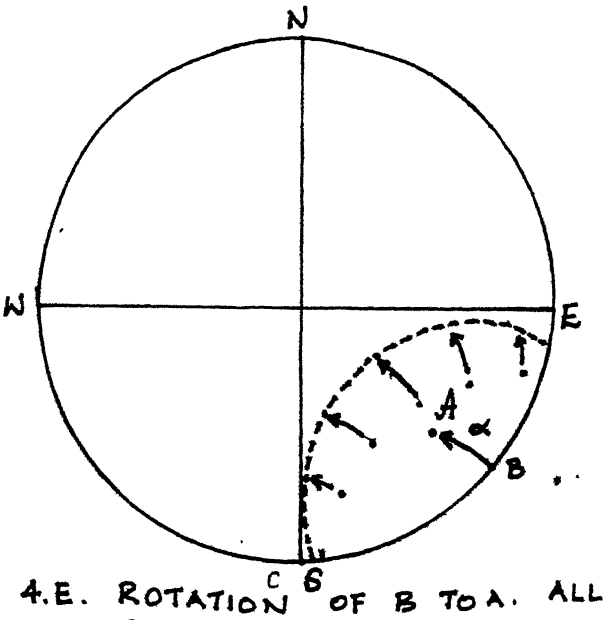
4.B. ROTATION OF B BY β DEGREES ABOUT THE PERPENDICULAR AXIS, TO C



4.C. LOCATING THE SMALL CIRCLE AT THE DESIR ANGLE FROM C



OTATION
ACK TO B
ROM C



4.E. ROTATION OF B TO A. ALL TH POINTS ARE ROTATED ABOUT N

axis. The arc containing the new position of these points is traced, which inturn will be at the requird number of degrees from the zone axis. This process is repeated for the next zone axis also. The intersection of the two small circles traced for each pole, fixes the location of the desired zone axis ([1 1 Ø]). Now the angle between this axis and the centre is measured by allignning the two poles to lie along the equator. In terms of this angle, the orientation of the crystal is expressed.

4.1.5. Optimising the distance :

The best estimate of the film to sample distance, D is determined by minimising the sum of suqares of the errors between the experimental angles ϕ_E and the theoretical angles ϕ_T, obtained with the final indices of the spots. The method adopted is the one described by Christensen et al. [5,6] and is as follows :

The mean square error, Z, is defined as,

$$Z(D) = \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} (\phi_E(D) - \phi_T)^2 \qquad (12)$$

where n is the number of spots indexed. The error is a function of the distance D. This error function is approximated by the quadratic,

$$Z(D) = a_{\emptyset} + a_1 D + a_2 D^2 \qquad (13)$$

The error is minimised with respect to D when,

$$D = D^* = \frac{-a_1}{2\,a_2}, \quad \text{if } 2a_2 > \emptyset. \qquad (14)$$

The co-efficients $a_{\emptyset}$, a_1, and a_2 are determined by a 3 point fit of the equations of the form :

$$Z_i = a_{\emptyset} + a_1 D_i + a_2 D_i^2 \qquad (15)$$

where i = 1,2, or 3 and Z_i is obtained by letting $D = D_i$. The initial values of D_1, D_2 and D_3 are taken as D, D - Ø.1 and D + Ø.1 cm respectively. With the standard value of 3 cm, this scheme provides an initial variation in D of ±1 mm. Substitution of the co-efficients $a_{\emptyset}$, a_1 and a_2 in equation 15, gives,

$$D^* = \frac{1}{2} \; \frac{Z_1(D_2^2 - D_3^2) + Z_2(D_3^2 - D_1^2) + Z_3(D_1^2 - D_2^2)}{Z_1(D_2 - D_3) + Z_2(D_3 - D_1) + Z_3(D_1 - D_2)} \qquad (16)$$

With the initial value of D as 3.Ø cm, a new value of D^* will be calculated using the expression 16. If these two values are within Ø.1 mm of each other, then D^* is taken as the optimised distance. Else, with D^* as D, expression 16 is used

to obtain a new value of D^*. This iterative procedure is continued until the two successive calculations are withim Ø.1 mm of each other. The best value of D obtained is used to calculate the optimized experimental angles.

The optimised distance is expected to be within 3.2 to 2.8 cm. If a value of best distance within this range is not obtained then, the scheme calculates the distance, within 3.2 to 2.8 cm which has got the least mean square error . The reason for putting such a restriction is explained in the next section.

4.1.6. Criteria for fixing the error limit :

The computer code is developed with an allowable error limit of $\pm$ 3.Ø degrees, in matching the interplanar angles. The error limit is set based on the errors likely to have been introduced during the measurement of input data. These are as follows :

(i) The spots on the X-Ray film generally do not have an exact location. The identification of the eaxct location is difficult, if the spots have fuzziness.

(ii) The experimental angles are calculated with the standard value of D as 3.Ø cm. But, while fixing the sample for X-Ray exposure, the distance of seperation between the sample and the X-Ray film could be any where between 2.8 to

3.2 cm. With sufficient care it will be possible to fix the distance within this range. Hence this limit is kept in optimising the distance of seperation. But since, the experimental angles are calculated initially with D as 3.Ø cm, there will a certain amount of error introduced in the experimental angles.

(iii) The X-Ray beam may not be passing through the centre of the hole punched in the film and hence the X and Y axes for the spots on the film may not be parallel to the edges of the film.

These errors can vary the measured experimental angles. The effect of X and Y values on the experimental angles is calculated. A set of 5 X and Y values were selected and they are varied 1.5 mm on both sides of the selected value. With a fixed value of D, the experimental angles are calculated for each value of X and Y. These angles are given in the Computer output No.1, in Appendix III. A maximum error of about 1.Ø degree is observed between the extreme ends of the X and Y values.

The effect of variation in experimental angles due to variation in D value is also calculated. For the same values of X and Y, experimental angles are calculated with the distance being increased in steps on 1 mm. These angles are given in computer output No.2 in Appendix III. Here a maximum

error of about 3.25 degrees is observed.

The error due to the X-Ray beam not passing through the centre of the punched hole is eliminated by fixing lead markers in the X-Ray film cassette. This fixes the X and Y axes of the film without ambiguity. This method has been tried for sample number 6.4 M.

Based on these calculation, the upper limit for the allowable error is kept at 3.Ø degrees.

4.2. Structure of the Program :

The computer code developed has been divided into number a of subroutines, each performing a specific task. The subroutines are arranged in the sequence, in which the tasks are to be performed. They are called from the main program in the same sequence. This chapter explains the function of each subroutine. The input data to be fed and their sequence is also explained. Flow cahrts are givan for each suroutine in Appendix IV.

4.2.1. Input Data :

The input data required are listed below in the same order in which they are fed.

1. Total number of spots to be indexed (N)

2. X and Y co-ordinates of each spot obtained from the X-Ray film, in cm
3. Total number of zones to be checked (NZ)
4. Numbers of spots belonging to each zone
5. Nutual angle between each zone.

Additional input data required are, error allowed in the angular match (ERROR), the maximum and minimum limits of the Miller indices to be assigned to the spots for index search (MAX and MIN), c/a ratio of the crystal (RATIO), the distance of seperation between the film and the sample (DIST). These data are given in the program itself, since they remain unchanged for all the samples.

A maximum of 15 spots can be indexed. This limitation is not absolute, but can be changed as required, by redefining the storage space, for the appropriate variables. The X and Y co-ordinates of each spot to be indexed should be obtained with an accuracy of $\pm$1.Ø mm. The procedure for obtaining the X and Y values has been explained in section 3.3. A stereogram of the pattern is to be prepared to identify the spots belonging to one zone. All the possible zones are identified and the zones which contain more than three spots are considered as important zones. Each zone is located on the stereogram and their mutual angles are measured. The spots are selected and numbered accordingly as explained in section 3.3.

The set of indices obtained is expected to be same for any order in which the spots are fed. But the number of iterations in each case may be different. For sample number 12.4, the spots are fed in different sequences and it is observed that the set of indices obtained remain the same, irrespective of the sequence.

4.2.1. Subroutine TRIANG(IA,IB) :

This subroutine calculates the angle between any two planes using their indices with the help of the crystallographic expression, for tetragonal crystals (eqn. 8). The counters IA and IB represent the spot numbers whose mutual angle is required. The latest indices assigned to the spots are used for calculating the angles. The c/a ratio value is transferred from the main program through COMMON statement. This routine is called from subroutine TEST, while the angular match is being carried out. The calculated angle is returned as TANGLE(IA,IB) representing the trial angle between the spot number IA and IB. When all the spots are indexed, this routine is called again from the main program and the mutual angle between the spots are measured and stored as theoretical angles. THis routine is not called from any other subroutine.

4.2.3. Subroutine EXPANG(D) :

This subroutine calculates the mutual angle between the

spots using their X and Y co-ordinates. The steps involved in calculating the angles are explained in section 4.1.1. The quantity D represents, the distance of seperation between the sample and the film. The angles can be measured for any given value of distance. This subroutine is called in the beginning from the main program with D as 3.Ø cm and the calculated angles are returned as EANGLE(I,J). These angles are stored as EANG(I,J) permanently, representing the experimental angles. THis subroutine is also called from subroutine ERRO, since the mutual angles are required for different values of distance D, to calculate the mean square erron bwteen the experimental and theoretical angles, during optimising the distance of seperation.

4.2.4. Subroutine ZANGL(MZN,IZ,ZANG) :

This subroutine calculates the angle between the zone axes, using the appropriate expression for the tetragonal crystal, (eqn. 9). This routine is called from subroutine ZONEM for matching the angles between the zone axes. The calculated angle is returned as ZANG, representing zone angle. The counters MZN, and IZ represent the numbers of the zones whose mutual angle is required. The indices of the zone axes are transferred through COMMON statements from the calling subroutine. The c/a ratio is transferred from the main

program. After the indices of all the spots are fixed, the zone angles are calculate using the final indices of the zone axes. This is carried out from the subroutine ZONE.

4.2.5. Subroutine SELECT :

The indices to be assigned for the spots is restricted between (5 5 5) to ($\bar{5}$ $\bar{5}$ $\bar{5}$). Hence all the possible indices which could be assigned to the spots are first selected within this range and they are stored permanently as IH,IK,IL. The selection of such a list is carried out in this subroutine. The selected indices are then rearranged from lower indices to higher indices based on the value of their d spacing. The starting index set is (Ø Ø Ø), which obviously will not be assigned to any spot. Three counters H,K,L are used in selecting the indices. The counter is first variedfrom Ø to the maximum limit in steps of 1. Whem L reaches the maximum limit, its value is changed to -1 and subsequently it is decreased in steps 1 upto to the minimum limit. After the counter L has been changed from +5 to -5, its value is reassigned as zero. This represents that the counter L has undergone one full cycle. Now the counter K is increased from zero to +1. With this value of K, the counter L again under goes one full cycle. Then K is increased in steps of 1 upto the maximum limit and for each change in K, L undergoes one

full cycle. When K completes one full cycle, (-5 to +5), the counter H is varied from -5 to +5, which is the last counter to be varied. With every change in counter H, both K and L have to undergo their respective cycles, to exhaust all possibile combinations of the Miller indices. Counter H needs to undergo only one cycle. When the values of H,K, and L reaches the minimum limit, it represents the end of the selection and all the counters are reassigned as zero. The total number of indices possible within this range are 11 x 11 x 11 - 1 = 133Ø. Certain index sets are eliminated in this range, as they either represent the same planes or are otherwise not permissible. Eliminations of such indices is carried out in the routine LAUE and the eliminations are explained in section 4.2.6. Each index setbefore being selected to be in the list is tested for these elimination conditions. After all the eliminations, the list contains a total of 578 index sets for (5 5 5) to ($\bar{5}$ $\bar{5}$ $\bar{5}$). The index set (Ø Ø Ø) is also included in the list, as the last entry.

The selected indices are rearranged from lower indices to higher indices. The larger the d spacing for a given index set, the lower will be the indices. On this basis, the d spacing for all the selected index sets are calculated and the index sets are rearranged in descending order of d spacing.

The indices from this list are assigned to the spots one

by one. The selected index sets are made available to the main program and the required subroutines.

4.2.6. Subroutine LAUE :

Each index set (to mean one combination of H,K and L), selected by the routine SELECT, is transferred to this routine to check, whether it is to be rejected by any of the elimination conditions. The elimination conditions checked are

(i) the sum (H + K + L), should be an even number.

The structure factor expression is,

$$F = \sum_{i=1}^{n} f_i e^{-2\pi i (hu + kv + lw)} \qquad (17)$$

where f is the atomic scattering factor, h,k,l are the Miller indices of the plane anf u,v,w are the atom positions in the unit cell of the crystal. For a body centered crystal the atom positions are Ø,Ø,Ø and 1/2,1/2,1/2. Hence the structure factor expression for a body centered crystal is,

$$F = f(1 + e^{-\pi i (h + k + l)}) \qquad (18)$$

$$\text{for } (h + k + l) = \text{even}, \; F = 2f \; , I \propto F^2 = 4f^2 \qquad (19)$$

$$\text{for } (h + k + l) = \text{odd}, \; F = Ø \text{ and } I = Ø \qquad (2Ø)$$

This implis that, there will be no reflection from planes with $h + k + l$ = odd. This is true for all boby centered crystals irrespective of the system to which they belong.

(ii) reduction of the indices to be tried can be achieved by noting that same value of angles result from all scalar multiples of an index set. Thus, if (1 1 Ø) is considered, then (2 2 Ø) or (3 3 Ø) need not be considered. In general if (h k l) is considered, then (nh nk nl) need not be considered, where n is an integer.

These conditions are selected for each index set selected and the decision is transferred through the logical variable EXTIN, to subroutine SELECT. If EXTIN is true, the particular set will not be included in the list and the selection procedure will continue to the next index set. This routine is called only from subroutine SELECT and not from any other routine.

4.2.7. Subroutine INDEX :

This subroutine does the 'Indexing' of the spots. Each spot is assigned with an index set which satisfies the interplanar angular relationship as well as zone relations. The spots are assigned indices from the list selected by the routine SELECT. The routine TEST is used for checking the angular match. Routine INDEX is called from the main program only once. All the input values are transferred to it through common statements.

The important variables used in this routine are,

(i) counter I; this represents the number of the spot to which the indices are assigned at any stage,

(ii) Logical variable ACCEPT; this takes the value true, if the indices assigned to the spot satisfy the angular match with all the other previously indexed spots. This variable is obtained from the routine TEST, since the angular match is checked in that routine,

(iii) Logical variable RANGE; if all the indices in the list have been tried for a particular spot, then the variable RANGE takes the value false. Thus, all the indices have been tried out for that spot and since no match is obtained, the indices of the previous spot has to be changed and the sequence of trying indices for the spot is repeated .

All spots are initially assigned the indices (Ø 1 1). The counter I decides at any stage which spot is being indexed. This subroutine has the provision, to see the intermediate values of the indices assigned upto a certain instant. Execution is paused after each 5ØØØØ iterations (this number can be changed), and the routine displays on the screen, the indices assigned to the spots upto this stage and asks if further execution is desired. If number 1 is keyed in, then the execution is continued and for any other numeral the execution is stopped and it comes to an abrupt end.

4.2.8. Subroutine TEST(J) :

This subroutine checks whether the angle between any two spots is within the allowable error limit (±3.Ø degrees). The counter J+1 represents the number of the spot which is undergoing the checking. The check is carried out using the routine TRIANG (see section 4.2.2), which calculates the interplanar angle with the indices assigned at any stage. This angle is then compared with the experimental angle between the corresponding spots, measured in the beginning from routine EXPANG. If the difference in the angles is within the allowable error limit, the indices assigned to the spots are accepted. The logical variable ACCEPT is used for this. Routine TEST also checks whether a given spot requires zone cheking and if so it decides whether the indices satisfy the zone condition. The subroutine ZONECK is called for checking the zone condition.

The indices assigned to a spot should satisfy the angular match with all the previous spots. The counters MN and MM are used for this check. The counter MN initially takes the value of J and MM is J+1. The value of MM is kept at J+1. Now the match between spotnumbers MN and MM is checked. If it is acceptable, MN is decreased by 1 (thus MN becomes J-1) and the angular match is checked between the current value of MN and MM. In this way, counter MN is

decreased upto 1. The indices assigned to spot number MM should satisfy the angular match with all the previous spots represented by the counter MN. Then only the logical variable ACCEPT takes the value true. If the match is not satisfactory at any stage then ACCEPT becomes false and the indices assigned to spot number MM should be changed.

Zone checking will be carried out only if ACCEPT is true. Zone checking is done only for specified spots already identified in the beginning. These spots are designated as zone points. When accept becomes true, the spot number MM is checked whether it is one of the zone points. This condition is determined by the logical variable ZONE. If ZONE is true, then the subroutine ZONECK will be called, in which the zone checking is carried out. If the zone condition is satisfied, the variable ZONEC becomes true. Depending upon the value of ZONEC, the variable ACCEPT finally takes the value true or false. If ZONEC is true, ACCEPT is also made true. If the final value of ACCEPT is true, it represents that the indices assigned to the spot MM (J+1), satisfies the angular match with all the previous spots and also the zone condition, if it is a zone point.

4.2.9. Subroutine ZONECK(M) :

This subroutine has to check two conditions,

(i) does the spot belong to the same zone as specified ?
(ii) if so, do the calculated values of the angle between various zone axes, match with the given values, within an error of $\pm$ 3.Ø degrees.

Condition (i) is checked in this routine itself and subroutine ZONEM is called for condition (ii). First the number zones to which the spot is common is determined. This is decided by the variable NZSB. Then for each zone, the condition that they should belong to the same zone is checked. If a given spot belongs to more than one zone, the condition (i) should be satisfied for all these zones. The decision is determined by the logical variable ZONEC.

Once condition (i) is satisfied, the calculated angles between zone axes should match with the given values obtained from the stereographic projection. This checking is required only if the spot belongs to zone numbers 2 or higher zone numbers. Hence, first the maximum number of the zone to which the spot belongs is determined. This is given by the variable MZN. Since angular match for zone axes is carried out in subroutine ZONEM, for MZN equal to 1, this subroutine will not be called. Otherwise, the zone axis of zone MZN is calculated first. The indices of zone axex are designated as M1(I),M2(I),M3(I), where I is the number of the zone. The counter IZ is used to number the other zone which is checked

with zone MZN. IZ initially takes the value MZN-1, and routine ZONEM is called. The decision is made by the logical variable ZONMAT. If ZONMAT is true, then IZ is reduced by 1, and the angukar match between zone IZ MZN is carried out. In this way IZ is reduced upto zone number 1. If at any stage ZONMAT becomes false, further checking is not carried out.

Depending upon the value of ZONMAT, the logical variable ZONEC is reassigned the value true or false. If ZONMAT is true, ZONEC is assigned the value true. ZONEC is then transferred to the calling routine TEST.

4.2.1Ø. Subroutine ZONEM(IZ)

The counter IZ has the same meaning as in the routine ZONECK. This routine ZONEM, first calculates the zone axis of the zone IZ. Then with these indices, the angular match between the zone IZ and MZN is checked. This is done by calling the routine ZANGL, which gives the value of the calculated zone angle as ZANG. This value is then compared with the given value for those particular zones. If the match is acceptable, ZONMAT becomes true. This routine is not called from any other subroutine.

4.2.11. Subroutine ADJUST(DI) :

This subroutine optimises the distance of seperation

between the sample and the X-Ray film. The initial distance is assumed to be 3.Ø cm. This distance is optimised by minimising the difference between the theroretical and experimental angles.

The scheme of optimising the distance is explained in section 4.1.4. The counter DI represents the initial value of the distance. The mean square error for this distance is initially calculated. The calculated error is stored as ZE. The distance is varied in steps of 1mm on both sides and the new value of distance is calculated. If the calculated value is within Ø.1 mm of the previous value, this new value is taken as the optimised distance. Else, the original value is changed to the new value of distance and the process is repeated.

A limit is set, within which the optimised distance is expected to be found. This limit is 2.8 cm to 3.2 cm. The reason for fixing this limit is explained in section 4.1.6. If the above scheme is not able to find the optimised distance within this range, then the program will select a distance within this range which has the least mean square error.

The logical variable FINAL is used to determine whether the distance range is exhausted or not. The variables DSTAR and ZERROR thakes the value of the optimised distance and the

error associated with that distance, respectively.

4.2.12. Subroutine ERRO(DK,DZ) :

This subroutine calculates the mean square error between the theoretical and experimental angles for a given value of distance using equation 12. The variables DK and ZK represent the value of distance and the mean square error respectively.

The value of experimental and theoretical angles are transferred from the calling routine ADJUST.

4.2.13. Subroutine ZONE :

After all the spots are indexed, the zone axes of each zone and the mutual angles of zone axes are calculated in this routine. This routine is called from the main program.

The zone axis is calculated using the indices of any two spots belonging to that zone. The indices of the zone axes are M1(I), M2(I), M3(I), where I is the number of the zone. The indices M1, M2, and M3 are given as,

$$M1 = (k_i * l_j - l_i * k_j) \quad (21)$$

$$M2 = (l_i * h_j - h_i * l_j) \quad (22)$$

$$M3 = (h_i * k_j - k_i * h_j) \quad (23)$$

where h_i, k_i, l_i and h_j, k_j and l_j are the indices of spots

i and j. The zone axes are calculated for all the zones.

The mutual angle between the zone axes are calculated by calling the subroutine ZANGL. The calculated angles are stored as ZANGLE(I,J) where I and J represent the zone numbers.

4.2.14. Main Program :

The main program first reads all the input values fed in the specified order, from the file 1N.1N. From the numbers of the spot belonging to one zone, the zone points are identified for each zone. Zone point is the number of the spot belonging to that zone, which is selected for carrying out the zone check. For each zone, three spot numbers are given. The maximum of these three numbers is taken as the zone point, for this zone. For example, if spots 2, 3 and 5 are belonging to one zone, spot 5 is taken as the zone point for that zone, because zone checking need not be carried out for spots 2 and 3. The counter ZP(I), represents the zone point for zone I.

After identifying the zone points, the zones are renumbered as 1,2,3 etc according to the zone points which are arranged in ascending order. The logical variables ACCEPT and RANGE, to be used later in the other subroutines, are given the initial value false and true respectively. All the

spots are given the intial indices (Ø 1 1).

The subroutine EXPANG(I,J) is called with the intial value of distance (3.Ø cm) and the experimental angles are stored as EANG(I,J) with I and J representing the spot numbers.

The main program calls the various routines in the order in which their tasks are to be performed. The routine INDEX is called first, since the indices of the spots are to be fixed first. INDEX transferrs the indices of the all the spots satisfying the required conditions. With these values the theoretical angles are calculated and the other subroutines ADJUST and ZONE are called. The optimised distance DSTAR is used finallly to calculate the experimental angles.

The subroutine PRINT is called finally which prints both the input and output values in a structured format.

CHAPTER 5. RESULTS AND DISCUSSIONS

Using the computer code developed, eight Laue back reflection patterns for Sn-Bi alloys were indexed and the orientation of each sample was determined, from the indices of the zone axes. The location of [1 1 Ø] direction is located on the stereographic projection for each sample and the angle between [1 1 Ø] and the centre is measured. The set of indices obtained for each sample from the computer program are given in Appendix III.

Table. 2 gives the results obtained from the computer program for each sample.

TABLE 2. RESULTS

Sample No.	Computer output No.	Number of spots indexed	Number of zones indexed	Maximum error in angles(deg)
2.1	3	5	2	2.5859
3.1	4	5	2	1.8539
4.3	5	5	2	2.2688
6.2	6	6	3	2.2354
6.3Ø	7	6	2	3.9867
6.4	1Ø	6	2	3.1596
6.4 M	8	5	2	Ø.8112
12.4	9	7	3	1.8656

Table 3. gives the orientation of each sample calculated from the stereographic projection of the pattern.

TABLE 3. ORIENTATION OF THE SAMPLES.

Sample No.	Indices of zone axes	Angle between [11Ø] and zone axes (deg)		Angle with [11Ø] and X-Ray beam (deg)
		measured	calculated	
2.1	[1 $\bar{1}$ 1]	89.Ø	9Ø.ØØ	54.Ø
	[3 $\bar{1}$ 1]	63.Ø	63.83	
3.1	[$\bar{1}$ Ø $\bar{3}$]	112.Ø	111.59	82.Ø
	[1 1 Ø]	Ø.Ø	Ø.ØØ	
4.3	[Ø 1 $\bar{1}$]	51.Ø	51.61	84.Ø
	[1 3 $\bar{3}$]	38.Ø	37.39	
6.2	[Ø $\bar{1}$ Ø]	134.Ø	134.95	49.Ø
	[3 $\bar{7}$ 1]	111.Ø	111.7Ø	
	[1 $\bar{1}$ 1]	9Ø.Ø	9Ø.ØØ	
6.3Ø	[$\bar{1}$ 1 $\bar{1}$]	89.Ø	9Ø.ØØ	76.Ø
	[$\bar{1}$ $\bar{1}$ 1]	158.Ø	158.84	
6.4 M	[1 1 $\bar{1}$]	22.Ø	21.Ø8	27.Ø
	[Ø 1 $\bar{1}$]	51.Ø	51.67	
6.4	[1 $\bar{5}$ $\bar{3}$]	122.5	121.83	not
	[$\bar{1}$ $\bar{1}$ $\bar{1}$]	159.5	158.84	known
12.4	[$\bar{1}$ 2 Ø]	7Ø.5	71.54	88.Ø
	[1 1 Ø]	Ø.Ø	Ø.ØØ	

The location of [1 1 Ø] direction has been determined on the stereographic projection, using the angle between the

zone axes, indexed by the program and [1 1 Ø]. The method of locating the [1 1 Ø] axis on the projection is explained in section 4.1.3.

The published [1,2,3] growth direction for pure tin and tin-rich bismuth alloys is [1 1 Ø]. Hence, this axis is located on the stereographic projection and the angle between this axsi and the centre of the projection is measured. It is observed from the results, that the [1 1 Ø] direction is not coinciding with the centre and the angular difference between [1 1 Ø] and centre is observed to be between 27.Ø degrees to 88.Ø degrees. The least angular difference is obtained for the sample 6.4 M (computer output no. 8, Appendix III). The X-Ray pattern of this sample has been obtained, after fixing lead markers in the inner cover of the film cassette. The maximum angular mismatch, for this sample is also found to be the least (Ø.81 degress).

In the light of these results obtained after fixing the lead markers in the film cassette, it may be worth while to take the LBR patterns for all the other experimental samples again.

It is clear that, the growth direction for the primary dendrites for the experimental samples of tin-rich alloys has not been found to be [1 1 Ø] as published in literature. The reason for this difference is not obvious at the moment.

Before concluding anything about the growth direction for the experimental samples, a series of confirmatory tests need to be taken. Such as,

(i) the scheme for indexing of the LBR patterns using computer should be modified, so that the zone axes are directly indexed rather than the spots.

(ii) another set of LBR patterns shuold be taken, in using the film cassette with the lead markers

(iii) specific grains need to be selected for LBR patterns atleast in some samples, which have sections in both the transverse and the longitudinal cut surfaces. This will permit the experimental measurement of the primary dendrite and its angle with thegrowth direction of the ingot, using optical microscope and micrographs.

Finally, as an independant confirmation for the crystallographic orientation of the samples, electron diffraction patterns can be taken using Transmission Electron Microscope.

CHAPTER 6. CONCLUSIONS

(i) A computer program in FORTRAN 77 has been developed for indexing Laue back reflection patterns for body centered tetragonal crystals. The program indexes the spots of the Laue pattern and their zone axes.

(ii) The computer code developed has been used for indexing eight Laue patterns obtained from directionally solidified Sn-Bi alloys. The crystallographic orientation of each sample has been determined.

(iii) The primary growth dirction for Sn and Sn-rich alloys which has been reported as [1 1 Ø], has not been confirmed for the experimental samples used in the study.

CHAPTER 7. SCOPE FOR FURTHER WORK

The present work carried out can be modified or extended in two stages,

(i) in the experimental work of preparing the samples and obtaining the Laue back rection patterns,

(ii) modification of the computer scheme employed.

7.1. Modifications in the experimiental work.

The samples require polishing and etching for X-Ray exposure. The tin-rich bismuth alloys used were at present polished in wjheel polishers using alumina as the polishing medium. Since these alloys are very soft, mechanical polisihng in some cases results in a distorted layer affecting the structure of the surface, which ultimately results in a ring pattern after X-Ray exposure. Alternatively, continuous polishing and etching using *chemois* leather has been tried and marked improvement in the quality of polishing is not observed. Electropolishing of the sample can be employed, which removesthe top layer without

affecting the structure. Since, a suitable set up was not available in the laboratory and within the available time frame it was not possible to fabricate one, this method is not tried.

The Laue film cassette requires markers for locating the X and Y axes on the film without ambiguity. Lead markers are provided in the inner cover of the cassette and an X-Ray pattern is obtained for sample no. 6.4 M only. This fixes the origin on the film clearly and hence the X and Y co-ordinates can be measured with considerable accuracy. This improved set up may be tried for other samples as well.

The X and Y co-ordinates of the spots were read using an ordinary graph paper with an accuracy of 1mm. A set up can be fabricated which has provisions for illuminating the pattern from the bottom andfor direct measurement of the X and Y co-ordinates. A set up similar to the one used for reading the lines of Debye-Scherrer patterns can be employed.

These modifications will considerably increase the confidence with which the input data are obtained, which ultimately will reduce error in angular mismatch and also the computer time.

7.2 Modifications of the computer scheme :

The present scheme first fixes he indices of the spots. Based on the indices of the spots, the indices of the zone axes are fixed. For determining the crystallographic orientation of the sample it will be enough to find out the indices of the zone axes alone. Hence, alternatively direct indexing of the zone axes could be employed.

The alternate scheme which could be employed is as follows:

From the stereographic projection, one or two spots which are lying at the intersection of atleast three zones shuold be indentified. Then three to four zone axes should also be identified with their mutual angles being measured. The input data will be the X and Y co-ordinates of the spots (if two spots are used), the angle between the zone axes. The scheme will assign indices to the spot(s) (which is expected to be of low indices). Then simultaneously the indices of the zones will also be fixed. The angular check will now be between a plane and a direction. The indices of the normal to a given plane for the tetragonal crystal can be obtained as follows,

$$\frac{h}{u} = \frac{k}{v} = \frac{l}{w}/(C/a)^2 \qquad (23)$$

With the indices of the plane being converted as the indices of a direction, the angle between the zone axes and the planes canbc measured, which will checked with the given values from the stereographic projection.

This scheme will require two lists of indices, one for the planes (spots) and the other for the directions (zones). The selection procedure used in the present scheme itself can be employed, for preparing these lists.

Modification of the current scheme to the above mentioned one is not carried out in this project due the time limitations. It will be worth while to try this scheme, since the indexing of the patterns by this scheme is expected to give interesting results.

CHAPTER 8. REFERENCES

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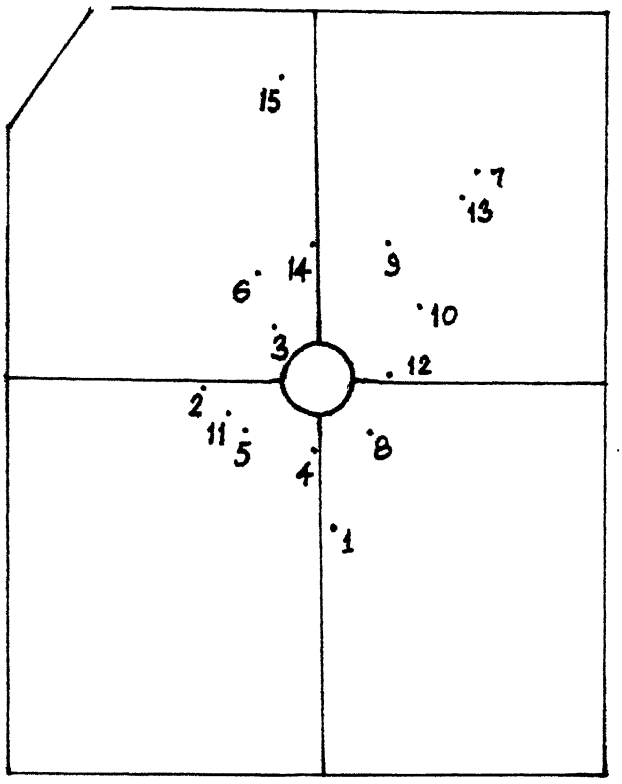
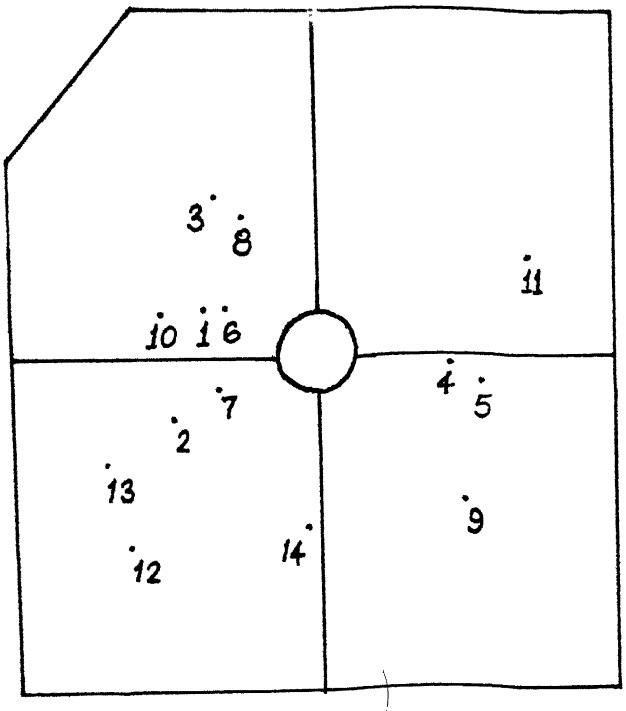
APPENDIX. I

LAUE FILMS (TRACINGS)



SAMPLE NO. 2.1

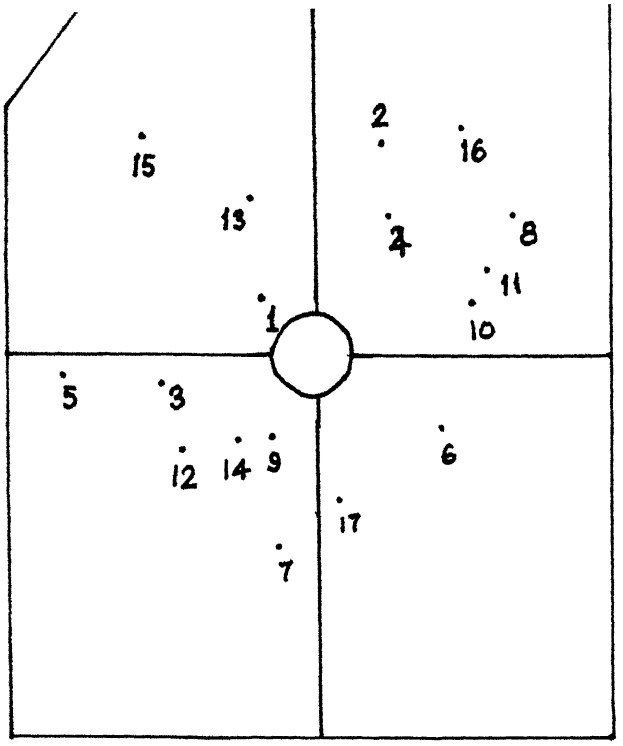
SPOT	X cm	Y cm
1	0.15	-1.95
2	-1.55	-0.55
3	-0.60	0.75
4	-0.05	-0.90
5	-1.00	-0.65
6	0.85	1.45
7	2.10	3.85
8	0.65	-0.65
9	0.85	1.90
10	1.35	1.05
11	-1.20	-0.40
12	0.90	0.10
13	1.90	2.50
14	-0.05	1.85
15	-0.15	3.15



SAMPLE NO. 3.1

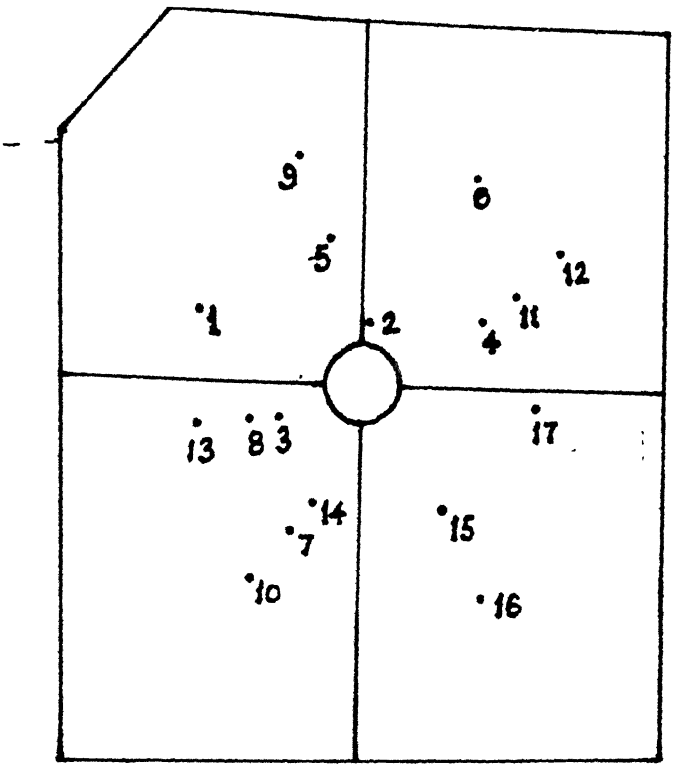
SPOT	X cm	Y cm
1	-1.45	0.45
2	-1.80	-1.05
3	-1.30	1.95
4	1.90	-0.15
5	2.30	-0.40
6	-1.15	0.50
7	-1.15	-0.60
8	-0.95	1.70
9	2.10	-2.00
10	-2.05	0.35
11	2.80	1.35
12	-2.30	-2.80
13	-2.65	-1.65
14	-0.20	-2.60

FIGURE I.2



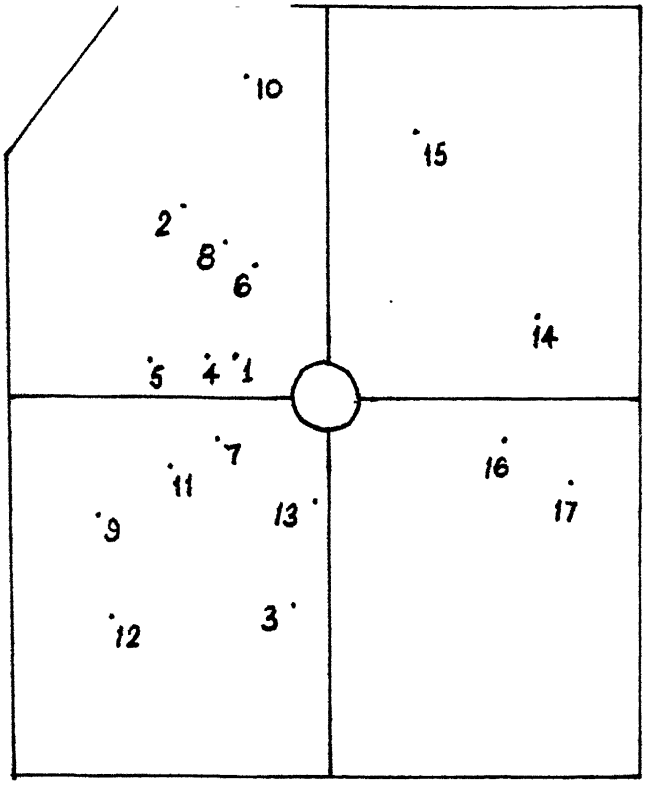
SAMPLE NO. 4.3

SPOT	X cm	Y cm
1	-0.80	0.80
2	0.90	1.80
3	-2.15	-0.45
4	1.00	1.80
5	-3.45	-0.35
6	1.70	-1.05
7	-0.60	-2.65
8	2.70	1.85
9	-0.65	-1.25
10	2.15	0.65
11	2.35	1.10
12	-1.90	-1.40
13	-0.85	1.95
14	-1.10	-1.25
15	-2.30	2.85
16	2.00	2.95



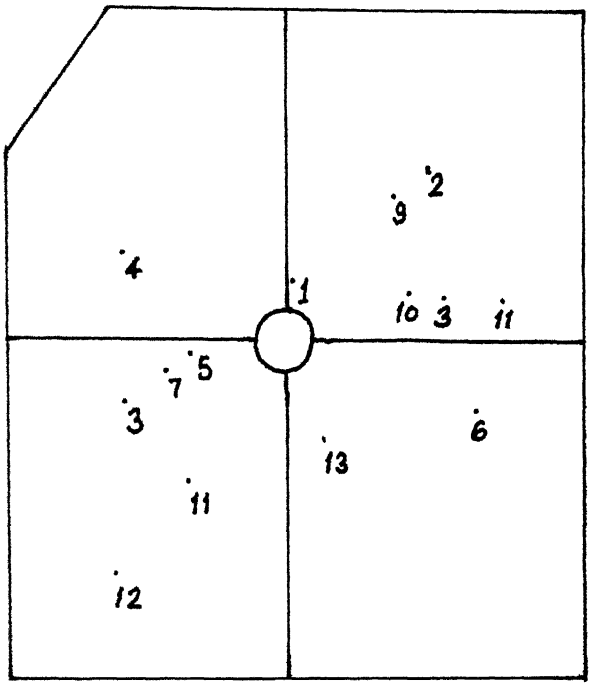
SAMPLE NO: 6.2

SPOT	X cm	Y cm
1	-2.25	0.85
2	0.00	0.80
3	-1.10	-0.54
4	1.75	0.90
5	-0.35	1.65
6	1.50	2.80
7	-0.95	-2.00
8	-1.55	-0.55
9	-0.95	2.95
10	-1.50	-2.65
11	2.10	1.25
12	2.65	1.75
13	-2.30	-0.62
14	-0.65	-1.65



SAMPLE NO. 6.30

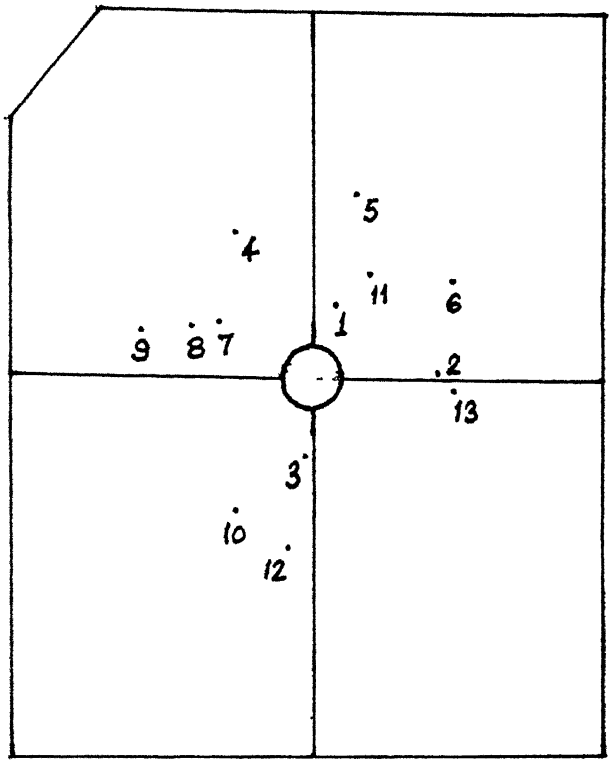
SPOT	X Cm	Y Cm
1	-1.30	0.40
2	- 1.95	2.35
3	- 0.45	- 2.95
4	- 2.40	0.30
5	- 1.65	0.35
6	- 1.00	1.60
7	- 1.50	0.75
8	- 1.40	1.90
9	- 3.10	- 1.75
10	- 1.15	4.15
11	- 2.10	- 1.15
12	- 2.75	- 3.15
13	- 0.15	- 1.65
14	2.80	1.05
15	1.15	3.45



SAMPLE NO. 6.4M

SPOT	X cm	Y cm
1	0.10	0.92
2	1.75	0.70
3	3.05	0.55
4	-2.25	1.20
5	-1.35	-0.05
6	2.65	-0.85
7	-1.65	-0.30
8	-2.20	-0.70
9	1.50	2.05
10	1.75	0.70
11	-1.35	-1.80
12	-2.35	-2.95

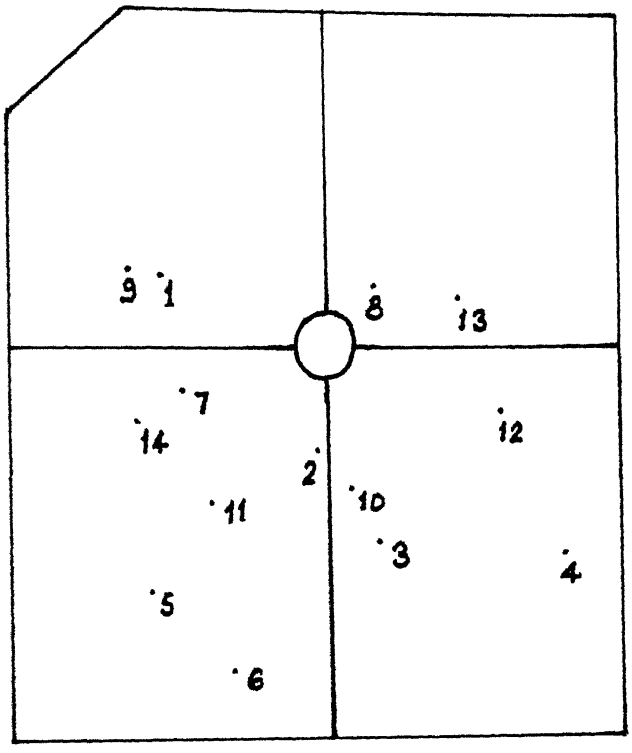
FIGURE. 1.6



SAMPLE NO. 12.4

SPOT	X cm	Y cm
1	0.35	0.90
2	1.75	0.05
3	-0.05	-1.15
4	-1.05	1.95
5	0.60	2.35
6	1.90	1.30
7	-1.25	0.68
8	-1.65	0.60
9	-2.30	0.55
10	-1.00	-1.85
11	0.80	1.35
12	-0.30	-2.35
13	1.95	-0.15
14	1.90	-1.70

FIGURE. I.7



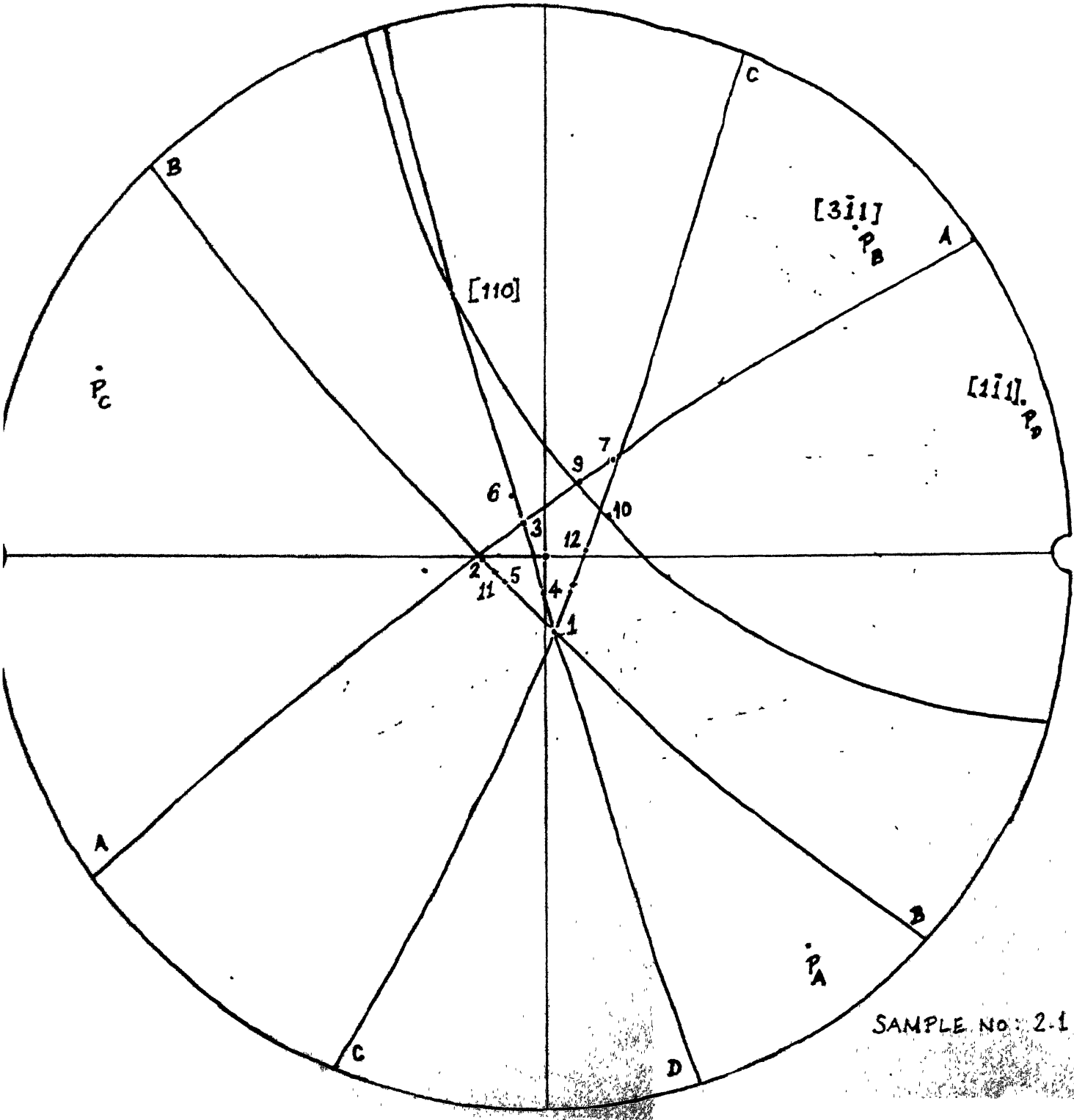
SAMPLE NO. 6.4

SPOT	X cm	Y cm
1	-2.15	1.05
2	-0.05	-1.35
3	0.70	-2.60
4	3.20	-2.80
5	-2.40	-3.20
6	-1.30	-4.30
7	-1.90	-0.50
8	0.75	0.80
9	-2.60	1.17
10	0.35	-1.90
11	-1.55	-2.10
12	2.40	-0.90
13	1.95	-0.65
14	-2.55	-0.95

FIGURE I.8

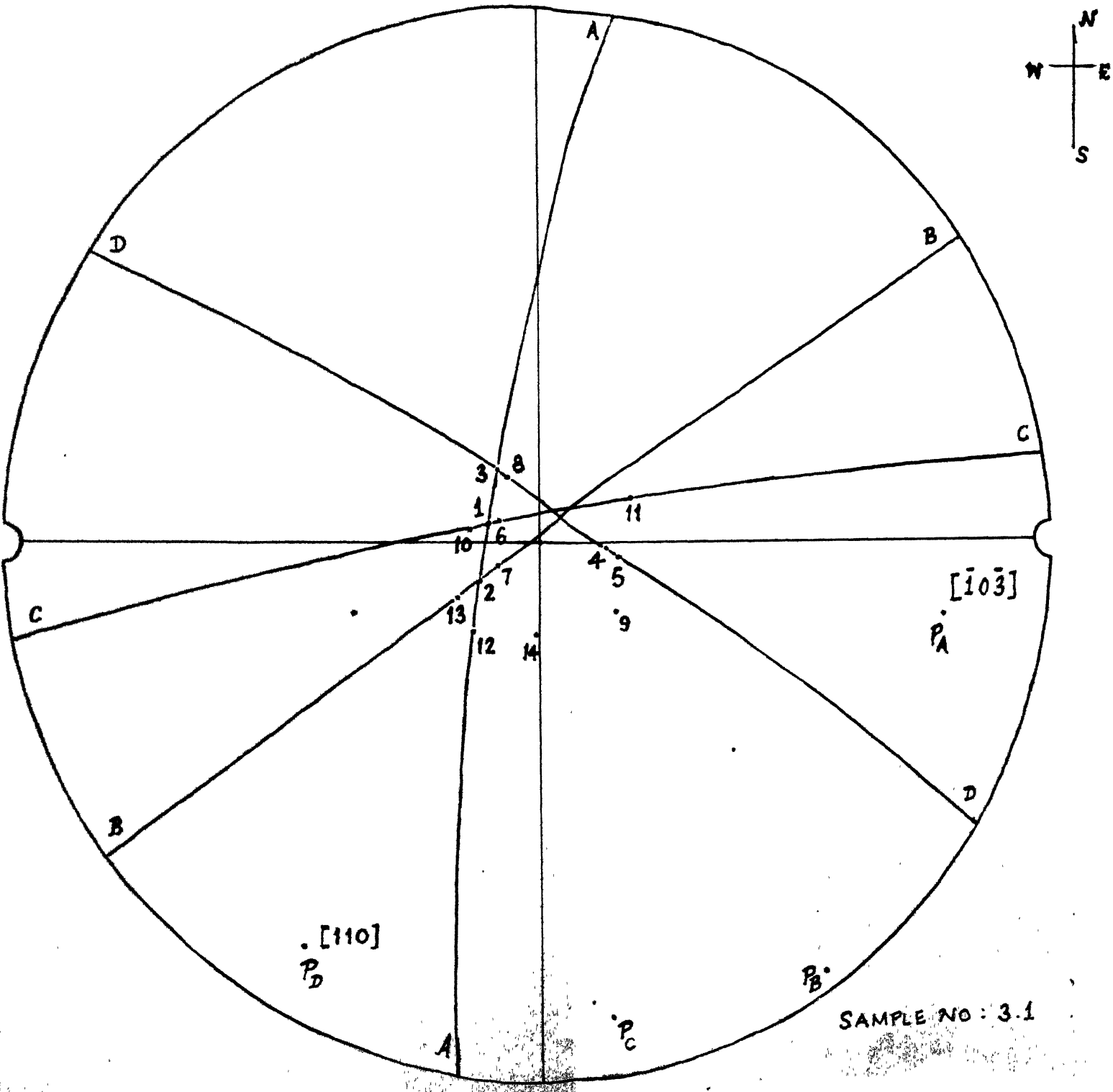
APPENDIX. II

STEREOGRAPHIC PROJECTIONS



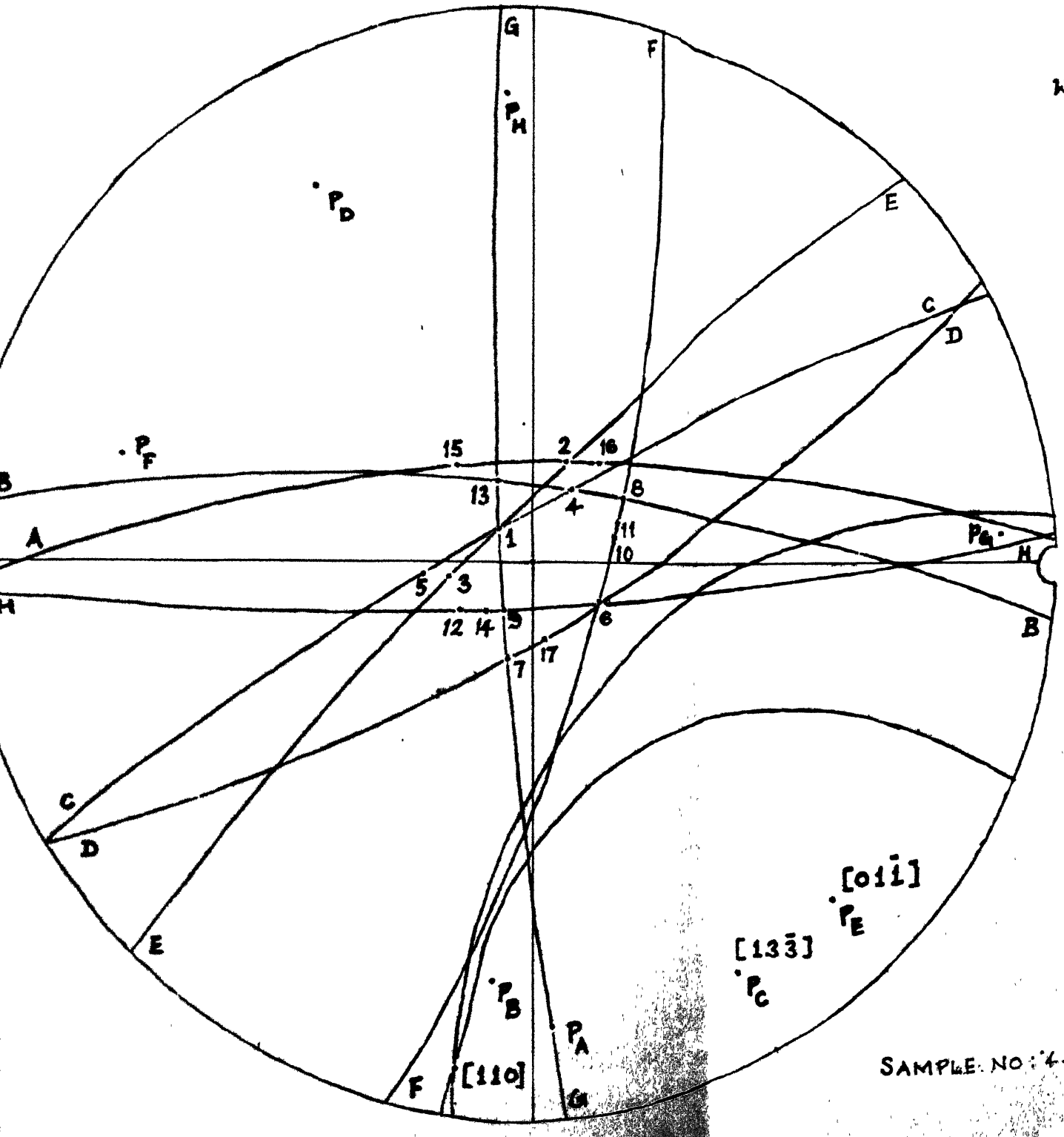
SAMPLE NO: 2-1

FIGURE II-1



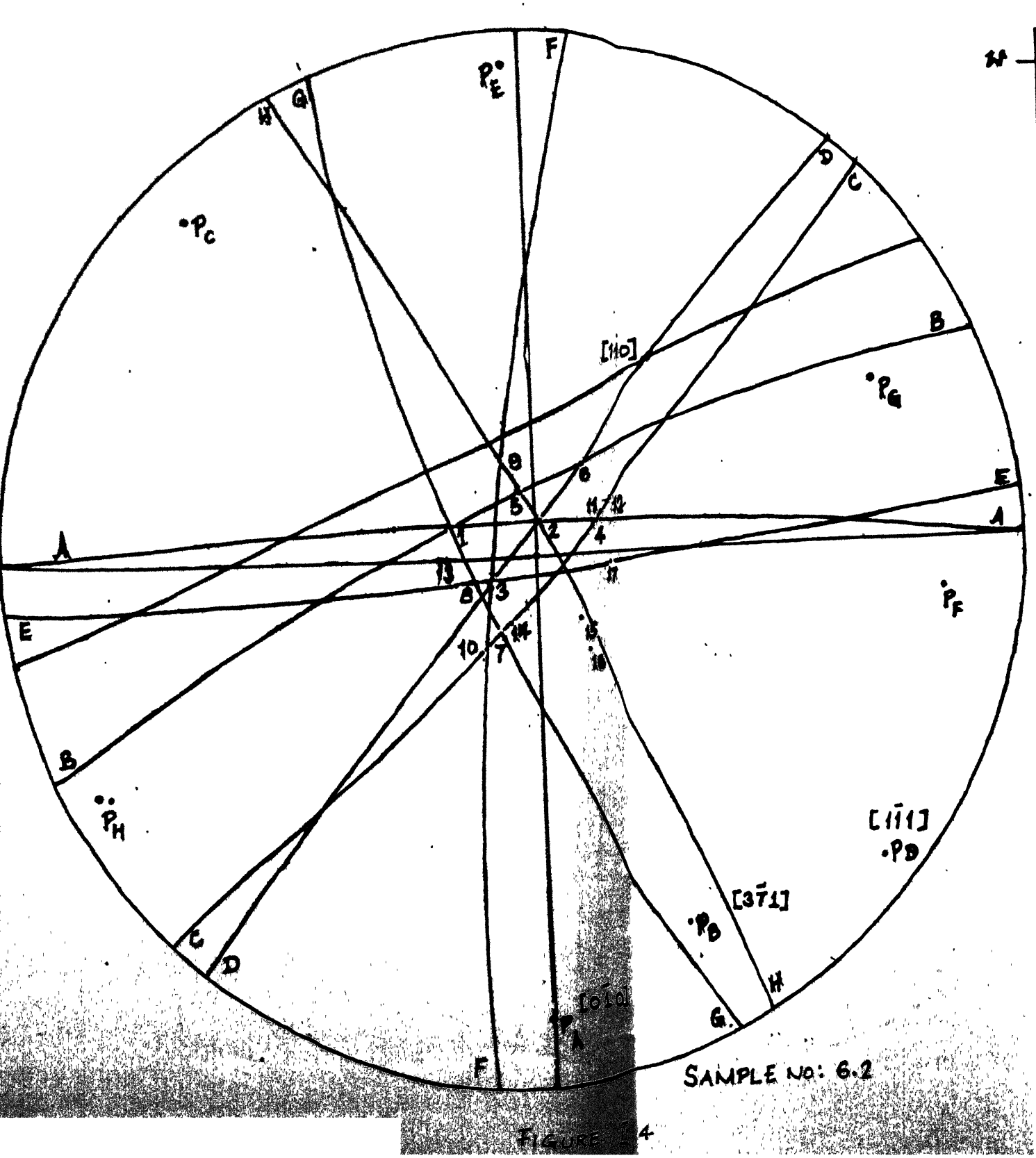
SAMPLE NO : 3.1

FIGURE II.2



SAMPLE NO: 4-3

FIGURE 4.3



SAMPLE NO: 6.2

FIGURE 4

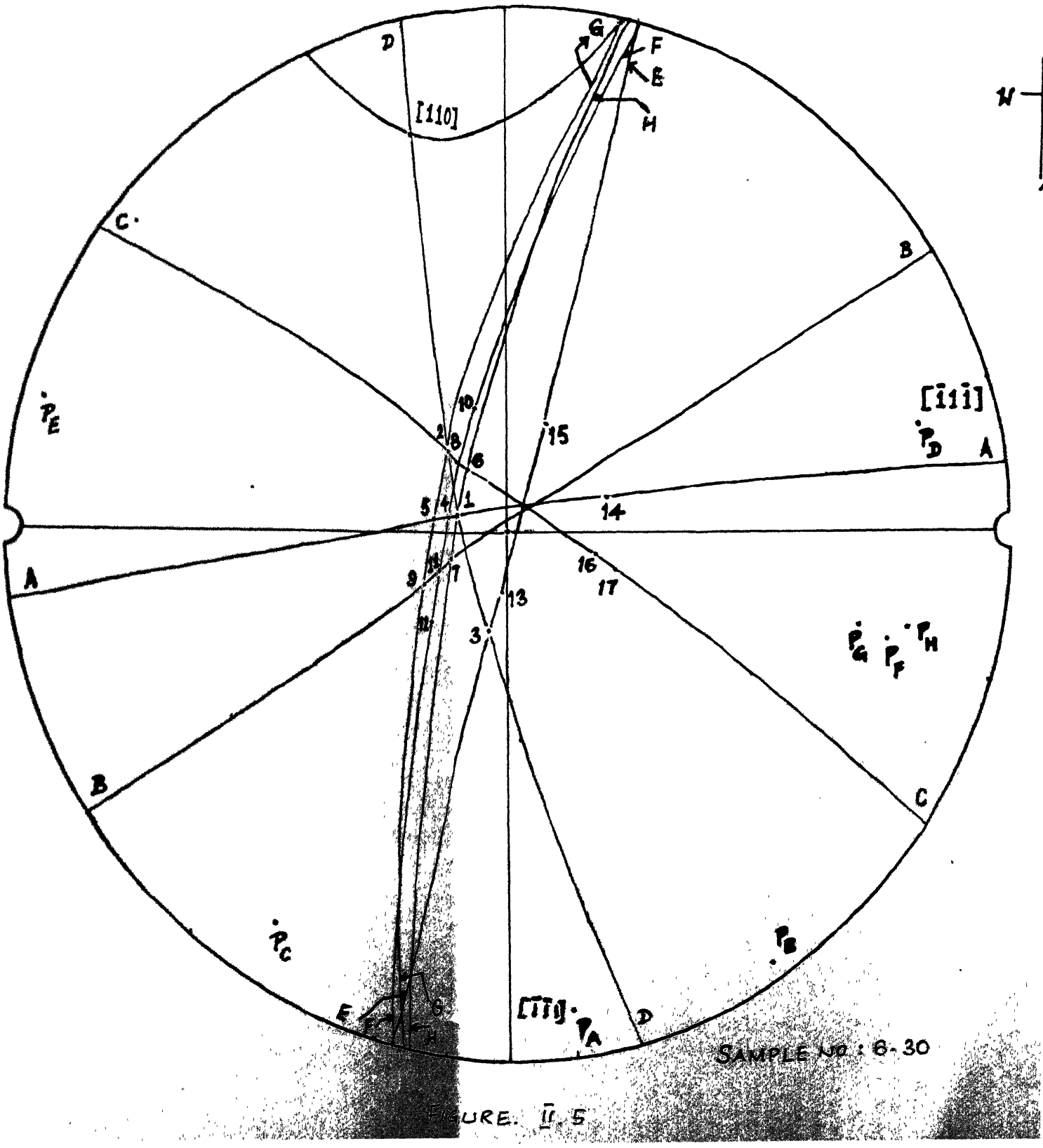

N
W
E
S
[110]
[1$\bar{1}$1]
[$\bar{1}$$\bar{1}$1]
A
B
C
D
E
F
G
H
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
SAMPLE NO : 6-30

FIGURE. II. 5

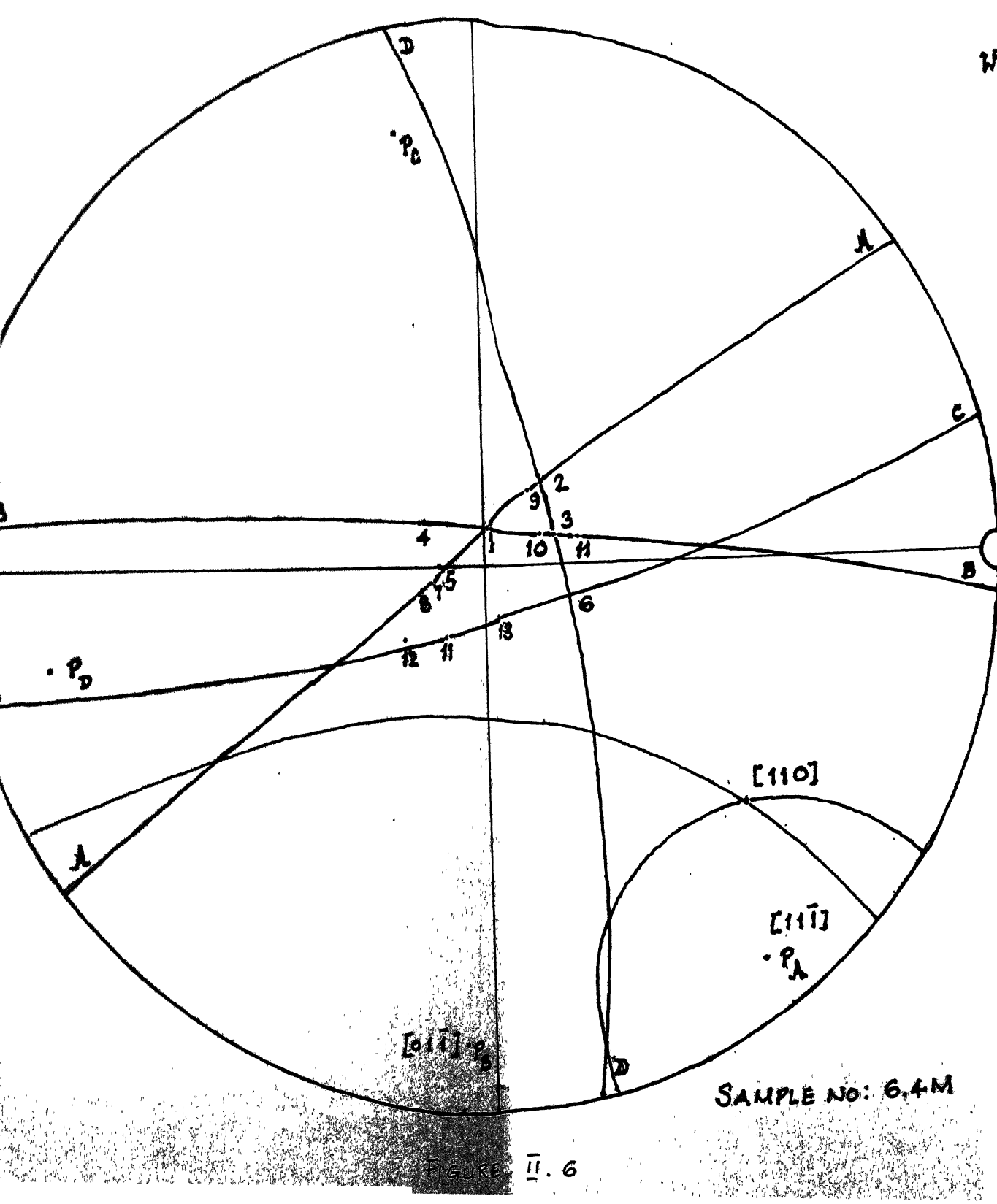

N
W
E
S
D
P_C
A
C
B
P_D
[110]
P_A
[011]
P_B
1
2
3
4
5
6
7
8
9
10
11
12
13
SAMPLE NO: 6.4M

FIGURE II. 6

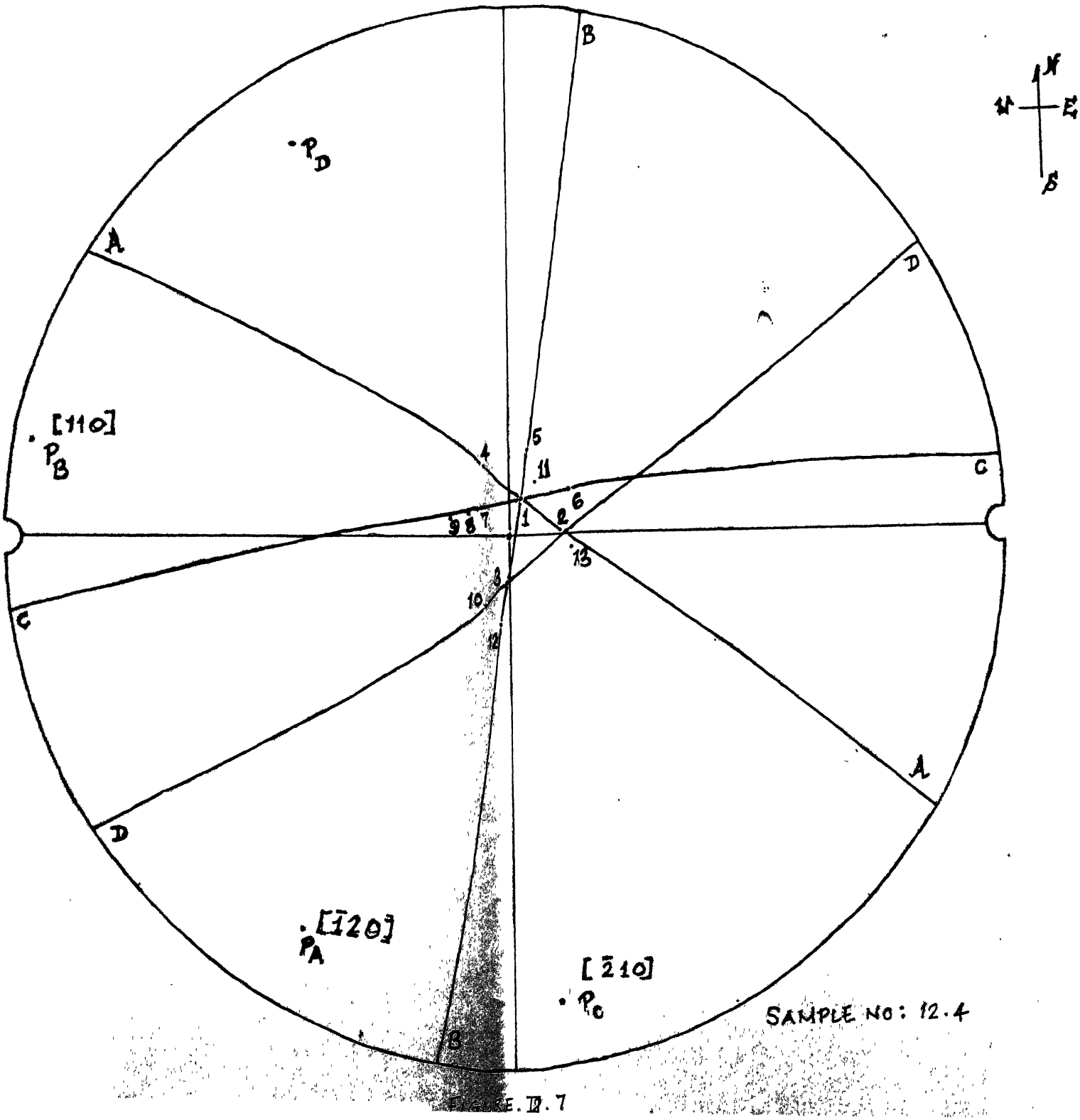
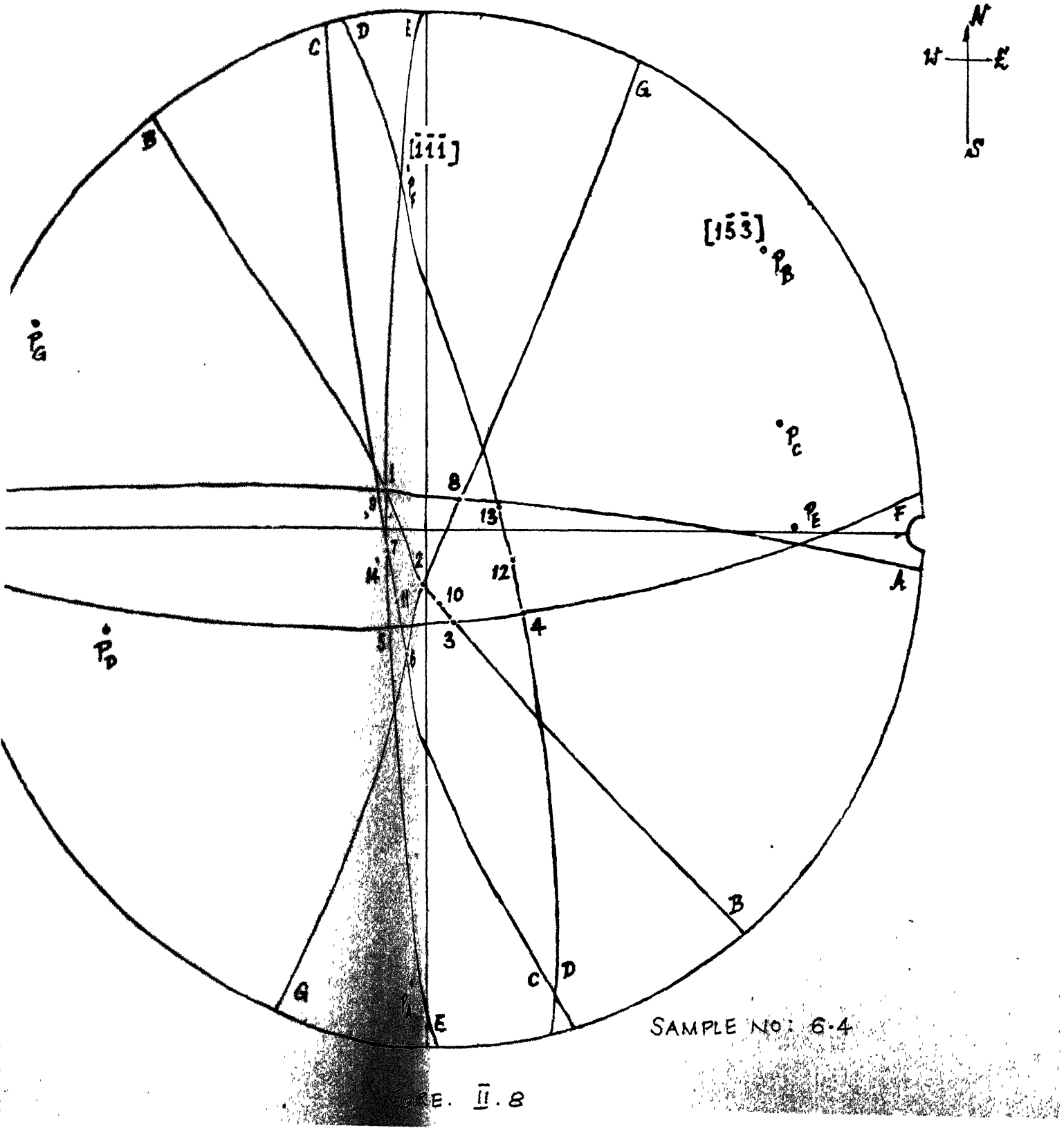
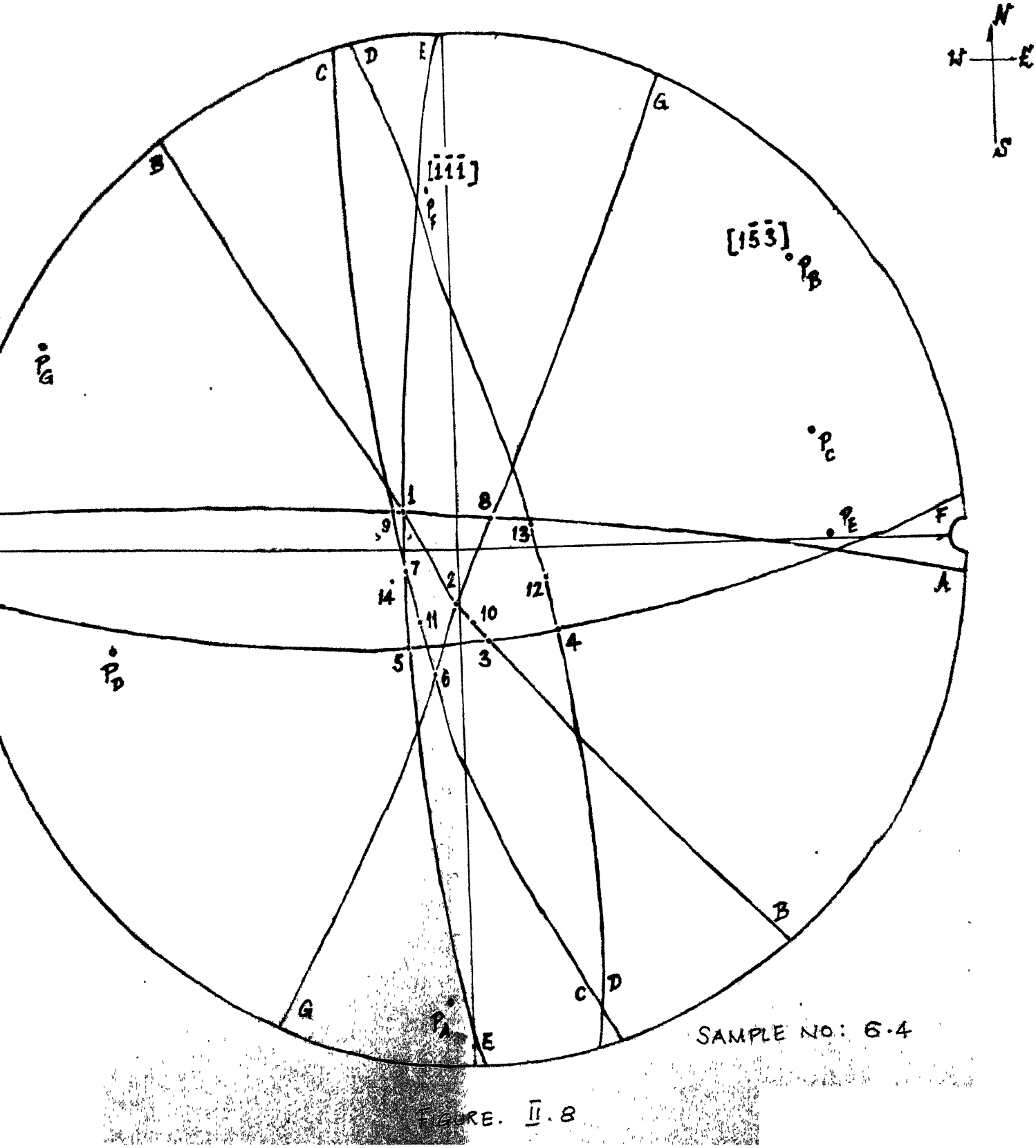

N
W
E
S
B
P_D
A
D
[110]
P_B
C
5
4
11
9
8
7
1
2
6
13
3
10
12
C
D
A
[$\bar{1}$20]
P_A
[$\bar{2}$10]
P_C
8
SAMPLE NO: 12.4

FIGURE. 12.7



SAMPLE NO: 6-4

FIGURE. II.8



SAMPLE NO: 6·4

FIGURE. II.8

APPENDIX. III

COMPUTER OUTPUTS

Number of points indexed = 5

Coordinates of the Points

(X)	(Y)
.15	-1.95
-1.55	-.05
-.60	.75
-.05	-.90
-1.00	-.65

The index search is between ($\bar{6}$ $\bar{6}$ $\bar{6}$) and (6 6 6)

Indices for Spot 1 = (0 1 1)

Indices for Spot 2 = ($\bar{1}$ 1 4)

Indices for Spot 3 = ($\bar{2}$ 1 3)

Indices for Spot 4 = ($\bar{1}$ 3 4)

Indices for Spot 5 = ($\bar{1}$ 2 5)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	21.42225	22.15983	22.66608	.73758	.50625
(1 3)	24.33289	26.63072	25.86430	2.29783	.76642
(1 4)	8.32377	9.59097	8.71459	1.26719	.87637
(1 5)	14.46566	17.37074	15.29183	2.90507	2.07891
(2 3)	10.78411	12.24081	11.45548	1.45671	.78533
(2 4)	15.20395	14.36950	16.18490	.83445	1.81540
(2 5)	6.95725	4.78908	7.37495	2.16817	2.58586
(3 4)	16.06454	17.03975	17.20922	.97521	.16947
(3 5)	13.17172	13.74046	14.06809	.56874	.32764
(4 5)	8.83872	9.93577	9.43303	1.09704	.50274

Number of points indexed = 5

Coordinates of the Points

(X)	(Y)
-1.45	.45
-1.80	-1.05
-1.30	1.95
1.90	-.15
2.30	-.40

The index search is between ($\bar{6}\ \bar{6}\ \bar{6}$) and (6 6 6)

Indices for Spot 1 = (3 $\bar{2}$ $\bar{1}$)

Indices for Spot 2 = (6 $\bar{2}$ $\bar{2}$)

Indices for Spot 3 = (6 $\bar{6}$ $\bar{2}$)

Indices for Spot 4 = (2 $\bar{2}$ $\bar{2}$)

Indices for Spot 5 = (5 $\bar{5}$ $\bar{6}$)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	12.45841	13.75203	11.96452	1.29362	1.78751
(1 3)	11.60867	10.83605	11.19025	.77262	.35420
(1 4)	29.42484	26.76682	28.09825	2.65803	1.33143
(1 5)	32.28025	31.34669	30.89296	.93355	.45373
(2 3)	24.02406	24.58804	23.11269	.56398	1.47535
(2 4)	31.87344	29.56003	30.51993	2.31341	.95990
(2 5)	33.81147	32.81430	32.44450	.99717	.36981
(3 4)	31.26182	28.96888	29.99240	2.29294	1.02352
(3 5)	34.39286	33.87995	33.04871	.51291	.83123
(4 5)	3.13190	4.91105	3.05714	1.77915	1.85391

mallest difference between the Angles = .3542

---

pots 1 2 and 3 belong to zone [$\bar{1}$ 0 $\bar{3}$]

---

pots 3 5 and 4 belong to zone [1 1 0]

	Theoritical	Exprimental
ngle between [$\bar{1}$ 0 $\bar{3}$] and [1 1 0] =	111.584396	109.000000

---

ptimised distance = 3.1708

ean square error in angles with Optimised Distance = 13.709590

OUTPUT NO. 4

Sample Number: 4.3 10. 7.1990

Number of points indexed = 5

Coordinates of the Points

(X)	(Y)
-.80	.80
.90	2.75
-2.15	-.45
1.00	1.80
-3.45	-.35

The index search is between ($\bar{6}$ $\bar{6}$ $\bar{6}$) and (6 6 6)

Indices for Spot 1 = (0 1 1)

Indices for Spot 2 = ($\bar{2}$ 3 3)

Indices for Spot 3 = (2 3 3)

Indices for Spot 4 = ($\bar{3}$ 6 5)

Indices for Spot 5 = (3 3 4)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	19.16835	17.69884	19.83531	1.46951	2.13647
(1 3)	14.82146	17.69884	15.43000	2.87739	2.26885
(1 4)	17.13297	15.96735	17.88799	1.16562	1.92064
(1 5)	19.32219	21.64357	19.85366	2.32138	1.78991
(2 3)	33.94245	35.39767	35.21635	1.45522	.18131
(2 4)	5.85726	5.01127	5.96423	.84599	.95296
(2 5)	37.50658	38.93271	38.67667	1.42614	.25604

)mallest difference between the Angles = .0804

)pots 1 3 and 2 belong to zone [0 1 $\bar{1}$]

)pots 4 1 and 5 belong to zone [1 3 $\bar{3}$]

	Theoritical	Exprimental
)ngle between [0 1 $\bar{1}$] and [1 3 $\bar{3}$] =	16.304283	14.500000

)ptimised distance = 2.8334

)ean square error in angles with Optimised Distance = 17.678785

OUPUT NO. 5

ımple Number: 6.2 5. 7.1990

Number of points indexed = 6

Coordinates of the Points

(X)	(Y)
-2.25	.85
.00	.80
-1.10	-.57
1.75	.90
-.35	1.65
1.50	2.80

The index search is between ($\bar{5}$ $\bar{5}$ $\bar{5}$) and (5 5 5)

Indices for Spot 1 = ($\bar{1}$ 0 3)

Indices for Spot 2 = ($\bar{1}$ 0 1)

Indices for Spot 3 = ($\bar{3}$ 2 5)

Indices for Spot 4 = ($\bar{5}$ 0 3)

Indices for Spot 5 = ($\bar{4}$ $\bar{1}$ 5)

Indices for Spot 6 = ($\bar{3}$ $\bar{1}$ 2)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
1) 2)	17.90457	18.30454	17.49496	.39997	.80959
1) 3)	14.13531	14.05289	13.83172	.08242	.22117
1) 4)	32.49332	31.96427	31.72059	.52905	.24368
1) 5)	16.12674	14.42539	15.77170	1.70135	1.34632
1) 6)	30.75963	31.13765	30.16601	.37802	.97164
2) 3)	16.00546	15.67426	15.53480	.33122	.13946

2	6)	17.01752	15.94853	16.74289	1.06899	.79436
3	4)	27.83989	26.68475	27.08817	1.15514	.40342
3	5)	20.43769	18.24898	19.88403	2.18871	1.63505
3	6)	33.01785	31.62279	32.27269	1.39506	.64991
4	5)	18.45053	19.51745	17.99938	1.06692	1.51807
4	6)	12.76141	12.27980	12.56005	.48161	.28025
5	6)	14.71627	16.71225	14.47686	1.99598	2.23539

---

argest difference between the Angles = 2.2354

mallest difference between the Angles = .1395

---

pots 1 2 and 4 belong to zone [0 $\bar{1}$ 0]

---

pots 1 6 and 5 belong to zone [3 $\bar{7}$ 1]

---

pots 2 6 and 3 belong to zone [1 $\bar{1}$ 1]

	Theoritical	Exprimental
ngle between [0 $\bar{1}$ 0] and [3 $\bar{7}$ 1] =	23.528664	24.000000
ngle between [0 $\bar{1}$ 0] and [1 $\bar{1}$ 1] =	48.703575	49.500000
ngle between [3 $\bar{7}$ 1] and [1 $\bar{1}$ 1] =	27.148540	28.000000

)ptimised distance = 3.0995

lean square error in angles with Optimised Distance = 15.232025

OUTPUT NO. 6

Sample Number: 6.3 5. 7.1990

Number of points indexed = 6

Coordinates of the Points

(X)	(Y)
-1.30	.40
-1.95	2.35
-.45	-2.95
-2.40	.30
2.80	1.05
2.35	-.65

The index search is between ($\bar{5}$ $\bar{5}$ $\bar{5}$) and (5 5 5)

Indices for Spot 1 = (0 1 1)

Indices for Spot 2 = (1 4 3)

Indices for Spot 3 = ($\bar{2}$ 1 3)

Indices for Spot 4 = (1 4 5)

Indices for Spot 5 = ($\bar{3}$ 5 2)

Indices for Spot 6 = ($\bar{3}$ 3 2)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	13.88614	11.16033	14.28382	2.72580	3.12349
(1 3)	26.44591	26.63072	27.52157	.18481	.89086
(1 4)	7.67693	7.60714	7.89340	.06980	.28626
(1 5)	32.75050	35.40897	34.18456	2.65847	1.22441
(1 6)	31.72933	33.87175	33.26652	2.14243	.60524
(2 3)	40.30534	37.79106	41.77771	2.51428	3.98666
(2 4)	14.44728	12.63699	15.12311	2.01029	2.48612

3	4)	27.82586	28.55518	28.83648	.72931	.28130
3	5)	37.47913	40.33776	38.82219	2.85863	1.51557
3	6)	26.45557	28.82194	27.40409	2.36636	1.41785
4	5)	40.40999	43.01610	42.05954	2.60611	.95657
4	6)	38.85129	40.87887	40.57034	2.02758	.30853
5	6)	12.25938	14.19532	12.65600	1.93594	1.53931

.argest difference between the Angles = 3.9867

:mallest difference between the Angles = .2813

:pots 1 2 and 3 belong to zone [$\bar{1}$ 1 $\bar{1}$]

:pots 4 5 and 1 belong to zone [$\bar{1}$ $\bar{1}$ 1]

	Theoritical	Exprimental
\ngle between [$\bar{1}$ 1 $\bar{1}$] and [$\bar{1}$ $\bar{1}$ 1] =	97.407150	95.500000

)ptimised distance = 2.8216

Mean square error in angles with Optimised Distance = 45.886284

OUTPUT NO. 7

ample Number: 6.4M 10. 7.1990

Number of points indexed = 5

Coordinates of the Points

(X)	(Y)
.10	.95
1.95	2.40
3.05	.55
-2.25	1.20
-1.35	-.05

The index search is between ($\bar{6}$ $\bar{6}$ $\bar{6}$) and (6 6 6)

Indices for Spot 1 = (0 1 1)

Indices for Spot 2 = ($\bar{1}$ 3 2)

Indices for Spot 3 = ($\bar{4}$ 5 5)

Indices for Spot 4 = (2 3 3)

Indices for Spot 5 = (2 3 5)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	16.10678	15.94569	15.81257	.16109	.13312
(1 3)	21.81234	20.95372	21.33066	.85862	.37695
(1 4)	18.43542	17.69884	17.98196	.73657	.28311
(1 5)	15.82153	15.66975	15.31705	.15178	.35270
(2 3)	14.45026	13.64662	14.18759	.80364	.54098
(2 4)	31.88161	31.43349	31.19319	.44812	.24031
(2 5)	31.91188	31.61542	31.11368	.29646	.50174
(3 4)	40.24323	38.65254	39.30821	1.59069	.65567
(3 5)	34.98332	34.25142	34.07983	.73190	.17159
(4 5)	11.19184	11.77154	10.96038	.57969	.81116

Smallest difference between the Angles = .1331

Spots 1 2 and 5 belong to zone [1 1 $\bar{1}$]

Spots 1 4 and 3 belong to zone [0 1 $\bar{1}$]

	Theoritical	Exprimental
Angle between [1 1 $\bar{1}$] and [0 1 $\bar{1}$] =	41.262600	39.000000

Optimised distance = 3.1112

Mean square error in angles with Optimised Distance = 2.083834

OUTPUT NO. 8

Sample Number: 12.4 5. 7.1990

Number of points indexed = 7

Coordinates of the Points

(X)	(Y)
.35	.90
1.75	.05
-.05	-1.15
-1.05	1.95
.60	2.35
1.90	1.30
-1.25	.68

The index search is between ($\bar{5}$ $\bar{5}$ $\bar{5}$) and (5 5 5)

Indices for Spot 1 = (0 0 2)

Indices for Spot 2 = (2 1 5)

Indices for Spot 3 = (1 $\bar{1}$ 2)

Indices for Spot 4 = ($\bar{2}$ $\bar{1}$ 5)

Indices for Spot 5 = ($\bar{1}$ 1 4)

Indices for Spot 6 = (1 2 5)

Indices for Spot 7 = ($\bar{1}$ $\bar{2}$ 5)

Error range allowed = 3.00 deg.

oints 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
1 2)	14.03932	13.70912	14.57744	.33020	.86832
1 3)	19.14144	21.09153	19.98469	1.95009	1.10684
1 4)	13.93702	13.70912	14.39327	.22790	.68415

(1 7)	14.36903	13.70912	14.96365	.65992	1.25454
(2 3)	18.74132	21.02935	19.47604	2.28803	1.55331
(2 4)	27.94303	27.41825	28.93628	.52478	1.51803
(2 5)	20.31370	19.99171	20.97113	.32198	.97942
(2 6)	9.69134	8.59591	9.98634	1.09543	1.39043
(2 7)	26.78162	25.98589	27.85150	.79573	1.86561
(3 4)	27.67121	28.42290	28.72496	.75169	.30206
(3 5)	29.80225	32.00727	30.91108	2.20502	1.09618
(3 6)	26.20103	28.42290	27.16849	2.22187	1.25441
(3 7)	19.53917	21.02935	20.35385	1.49018	.67550
(4 5)	12.89019	14.50506	13.27177	1.61487	1.23328
(4 6)	23.70345	25.98589	24.45648	2.28244	1.52941
(4 7)	9.97087	8.59591	10.27745	1.37497	1.68154
(5 6)	12.61763	14.50506	12.98671	1.88743	1.51834
(5 7)	19.86813	19.99171	20.52667	.12358	.53496
(6 7)	26.62463	27.41825	27.58488	.79362	.16663

Largest difference between the Angles = 1.8656

Smallest difference between the Angles = .0127

Spots 1 2 and 4 belong to zone [$\bar{1}$ 2 0]

Spots 1 3 and 5 belong to zone [1 1 0]

Spots 1 6 and 7 belong to zone [$\bar{2}$ 1 0]

	Theoritical	Exprimental
Angle between [$\bar{1}$ 2 0] and [1 1 0] =	71.536263	70.000000
Angle between [$\bar{1}$ 2 0] and [$\bar{2}$ 1 0] =	36.855064	39.500000
Angle between [1 1 0] and [$\bar{2}$ 1 0] =	108.391327	109.000000

Optimised distance = 2.8626

Mean square error in angles with Optimised Distance = 28.823238

OUTPUT NO.9

Sample Number: 6.4 5. 7.1990

Number of points indexed = 6

Coordinates of the Points

(X)	(Y)
-2.15	1.05
-.05	-1.35
.70	-2.60
3.20	-2.80
-2.40	-3.20
-1.30	-4.30

The index search is between ($\bar{5}$ $\bar{5}$ $\bar{5}$) and (5 5 5)

Indices for Spot 1 = (2 1 $\bar{1}$)

Indices for Spot 2 = (3 3 $\bar{4}$)

Indices for Spot 3 = (1 2 $\bar{3}$)

Indices for Spot 4 = (0 1 $\bar{1}$)

Indices for Spot 5 = (4 1 $\bar{5}$)

Indices for Spot 6 = (2 1 $\bar{5}$)

Error range allowed = 3.00 deg.

Points 1	Expt.Ang 2	Thert.Ang 3	Optm.ang 4	Diffrnce (3-2)	Opt.Diff (4-3)
(1 2)	26.22742	23.61907	25.14823	2.60834	1.52916
(1 3)	36.01490	34.87347	34.68253	1.14143	.19094
(1 4)	46.15816	43.75464	44.67071	2.40351	.91607
(1 5)	27.86072	27.38269	27.10386	.47803	.27883
(1 6)	34.70551	36.93509	33.77548	2.22958	3.15961
(2 3)	9.92065	11.25439	9.66200	1.33374	1.59238

(2 6)	16.59824	17.53871	16.41047	.94048	1.12824
(3 4)	13.99994	12.95079	13.73789	1.04915	.78710
(3 5)	19.54600	19.02359	19.07720	.52241	.05361
(3 6)	13.87253	13.74046	13.64897	.13207	.09148
(4 5)	33.35070	31.97440	32.61627	1.37631	.64187
(4 6)	26.53683	25.35882	26.01917	1.17801	.66035
(5 6)	8.30994	11.21263	8.16258	2.90268	3.05005

Largest difference between the Angles = 3.1596

Smallest difference between the Angles = .0536

Spots 3 2 and 1 belong to zone [1 $\bar{5}$ $\bar{3}$]

Spots 4 5 and 3 belong to zone [$\bar{1}$ $\bar{1}$ $\bar{1}$]

	Theoritical	Exprimental
Angle between [1 $\bar{5}$ $\bar{3}$] and [$\bar{1}$ $\bar{1}$ $\bar{1}$] =	52.909092	55.000000

Optimised distance = 3.1700

Mean square error in angles with Optimised Distance = 33.340988

OUTPUT NO.10

APPENDIX. IV

LIST OF VARIABLES

LIST OF VARIABLE NAMES

NAME	TYPE	REMARK
1. ACCEPT	: Logical	Decides whether the indices assigned to any two spots are within the error limit
2. ALLOW	: Logical	Decides whether an index selected for a particular spot is allowbale.
3. DIFF(I,J)	: Real, Subscripted	Diference between EANG and TANGLE values The counters I and J represent the spot numbers.
4. DSTAR	: Real	Optimised value of the distance of seperation
5. DIST	: Real	Distance of seperation between the sample and the X-Ray film.
6. EANG(I,J)	: Real, Subscripted	Mutual angle between the Spots obtained from their X and Y coordinates and the initial distance. The counters I,J represent the Spot numbers.
7. EANGLE(I,J)	: Real, Subscripted	Mutual angle between the Spots obtained from their X and Y coordinates and the optimised distance. The counters represent the Spot numbers.
8. ERROR	: Real	Allowable angular deviation
9. EXTIN	: Logical	Decides whether an index satisfies the extinction conditions.
10. FINAL	: Logical	Limit within which the optimised value of distance to be found out. (2.5 to 3.5 cm)
11. FINISH	: Real	difference between the distances of any two consecutive iterations during optimisation.
12. H(I),K(I),L(I)	: Integer, Subscripted	Indices of the Spots. The counter I represents the Spot number.

13. HDIFF	: Real	Largest difference between EANG and TANGLE values.
14. IDATE	: Integer	Date of execution.
15. IH(I),IK(I),IL(I)	: Integer, Subscripted	Trial indices selected for the spots. The counter I represents the Spot number.
16. IP(I,J)	: Integer, Subscripted	Numbers of the Spots belonging to one zone. The counters I and J represent the numbers of the Spots belonging to ine zone.
17. J(I)	: Integer, Subscripted	Counter used to repres -ent the position of its index in the list. The counter represent the Spot number.
18. M1(I),M2(I),M3(I)	: Integer, Subscripted	indices of Zone axis. The counter I represent the zone number.
19. MAX	: Integer	Maximum limit of the Index search
20. MIN	: Integer	Minimum limit of the Index search
21. MONTH	: Integer	Month of execution.
22. MZN	: Integer	The maximum number of the zone to which the spot belongs.
23. N	: Integer	Total Number of Spots to be indexed.
24. NERRO	: Integer	Number of times the subroutine ERRO is called.
25. NEXP	: Integer	Number of times the subroutine EXPANG is called.
26. NIND	: Integer	Number of iterations carried out in the subroutine INDEX.
27. NLAUE	: Integer	Number of times the subroutine LAUE is called.
28. NTEST	: Integer	Number of times the subroutine TEST is called.
29. NTRI	: Integer	Number of times the subroutine TRIANG is called.
30. NTRIAL	: Integer	Number of times the subroutine TRIAL is called.

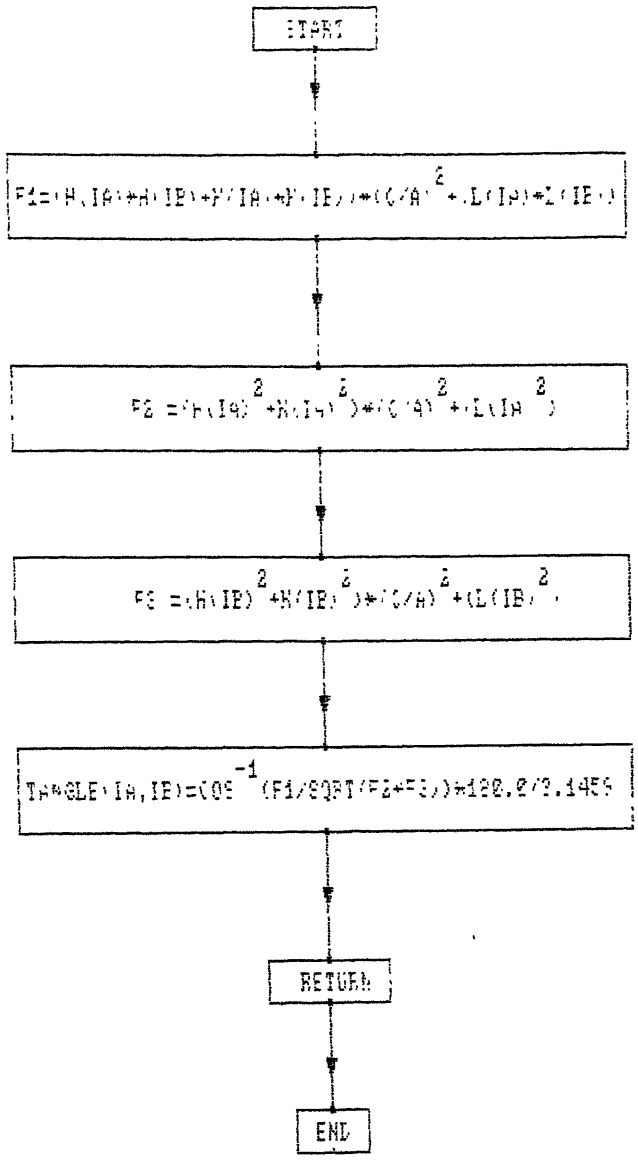
31. NZ	: Integer	Total number of zones.
32. NZM	: Integer	Number of times the subroutine ZONEM is called.
33. NZONE	: Integer	Number of times the subroutine ZONECK is called.
34. NZP	: Integer	Number of zone points.
35. NZSB	: Integer	Total number of zones to which a spot belong
36. RANGE	: Logical	Decides whether the index selected for a spot is within the range fixed.
37. RATIO	: Real	(C/a) Ratio of Pb-Bi.
38. SAMPLE	: Real	Sample number.
39. SDIFF	: Real	Smallest difference between EANG and TANGLE values.
40. TANGLE(I,J)	: Real, Subscripted	Mutual angle between the Spots obtained from their indices using the crystallo -grphic expression
41. X(I),Y(I)	: Real, Subscriptd	X and Y coordinates of the Spots on the film.
42. ZANG	: Real	Mutual angle between any two zone axes.
43. ZANGLE(I,J)	: Real, Subscripted	Mutual angle between the zone axes obtained after all the spots are indexed.
44. ZERROR	: Real	Mean square error between EANGLE and TANGLE values.
45. ZONANG(I,J)	: Real, Subscripted	Mutual angle between zone axes, obtained from the stereographic projection.
46. ZONE	: Logical	Decides whether zone checking is required for a particular spot.
47. ZONEC	: Logical	Decides whether a set of spots belong to the same zone.
48. ZONMAT	: Logical	Decides whether the mutual angle between the zone axes match with the given values.

49. ZP(I)	: Real, Subscripted	Zone point.
50. ZSB(I)	: Integer, Subscripted	Number of the zone to which a spot belongs.

APPENDIX. V

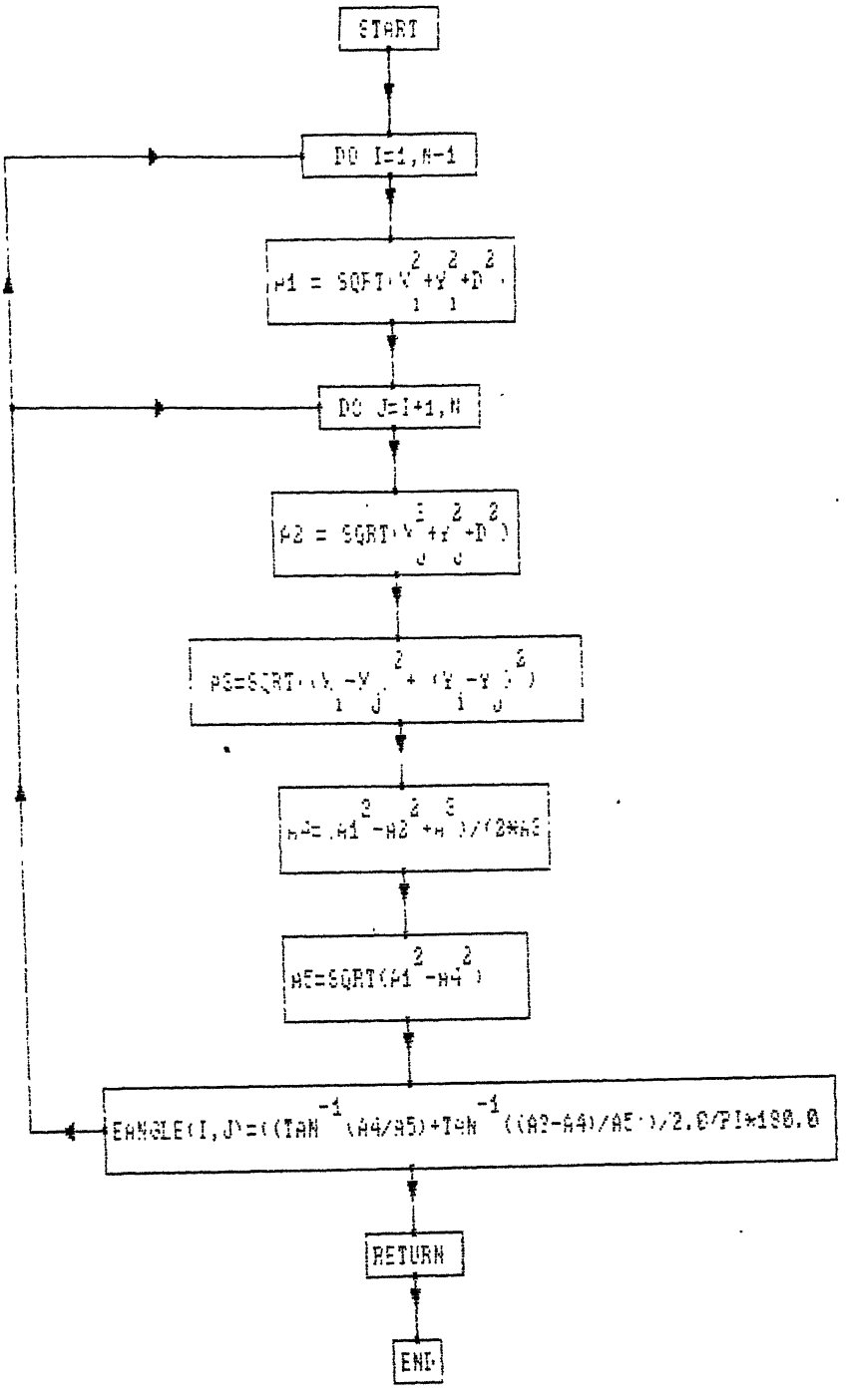
FLOW CHARTS

SUBROUTINE TRIANG(IA,IB)



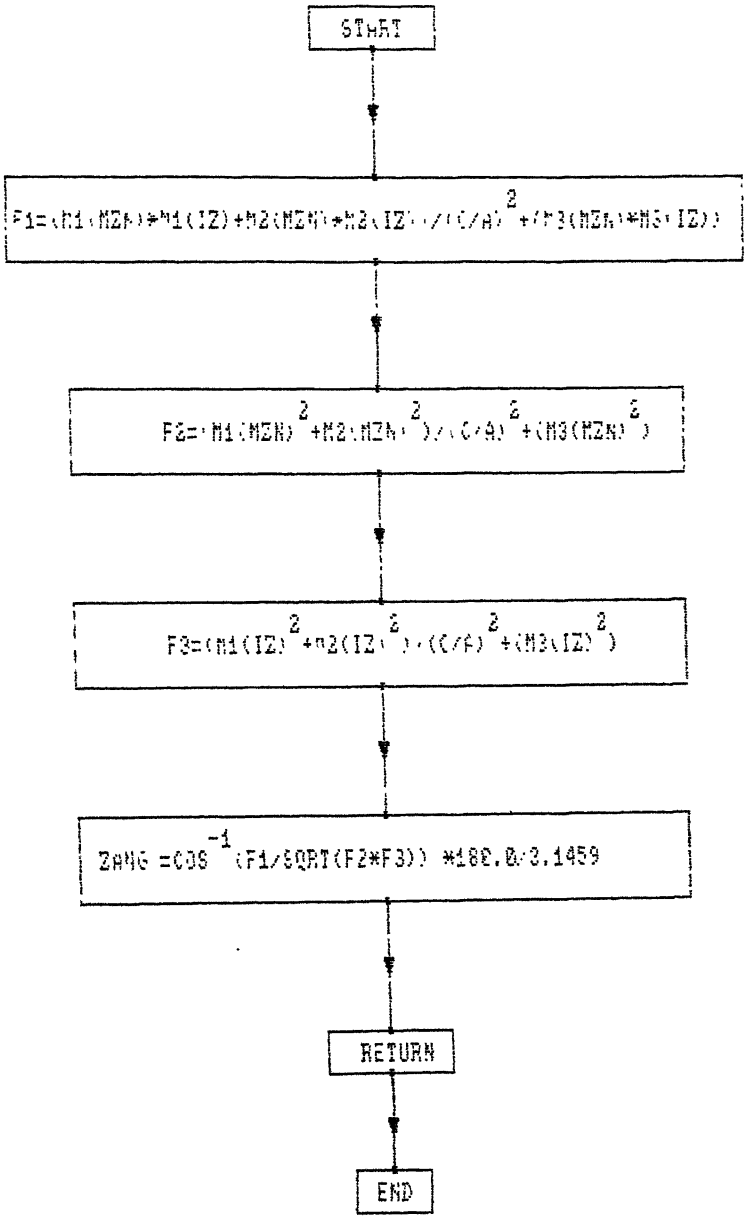
FLOW CHART NO: 1.

SUBROUTINE EXPANG(D)



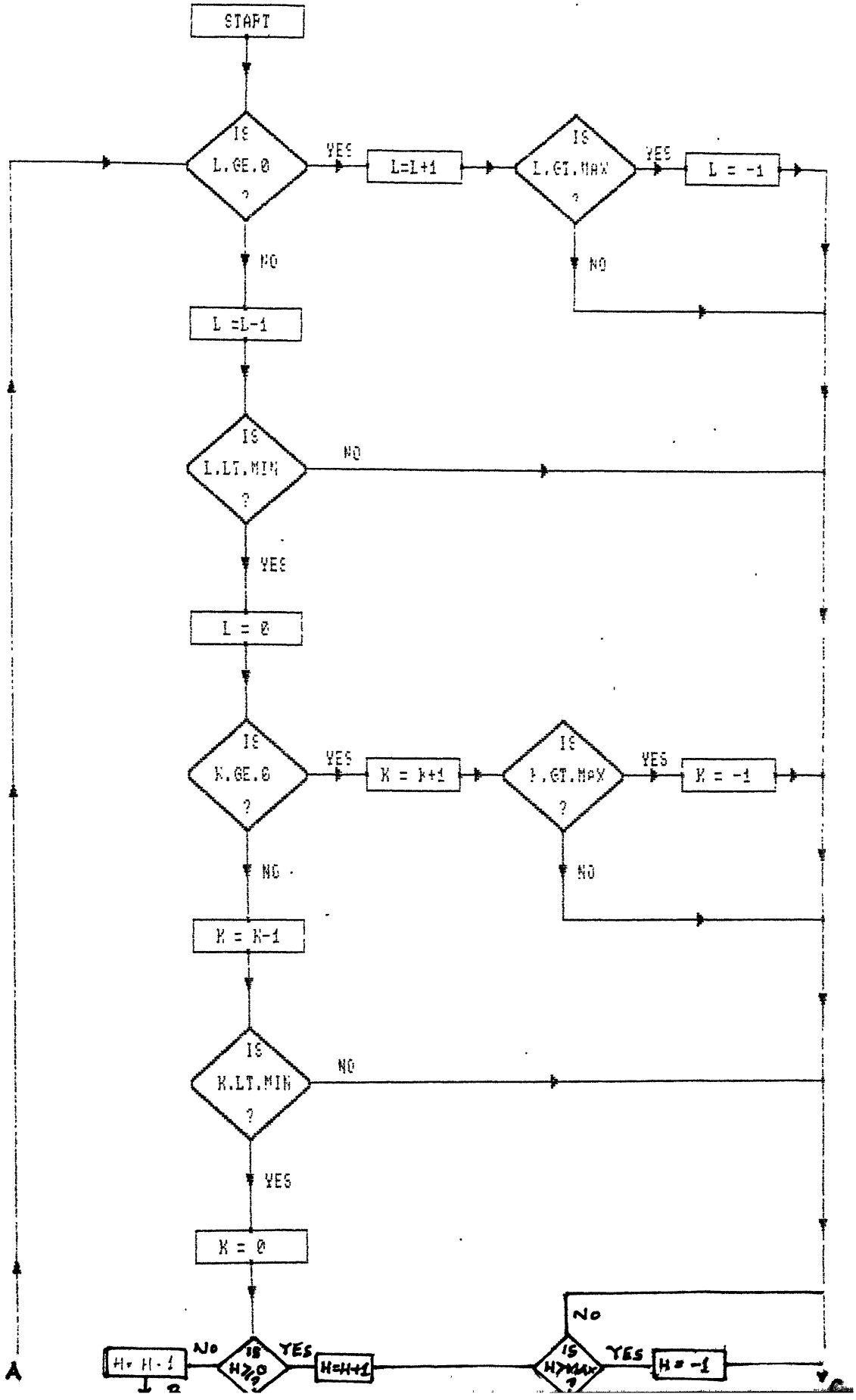
FLOW CHART NO. 2

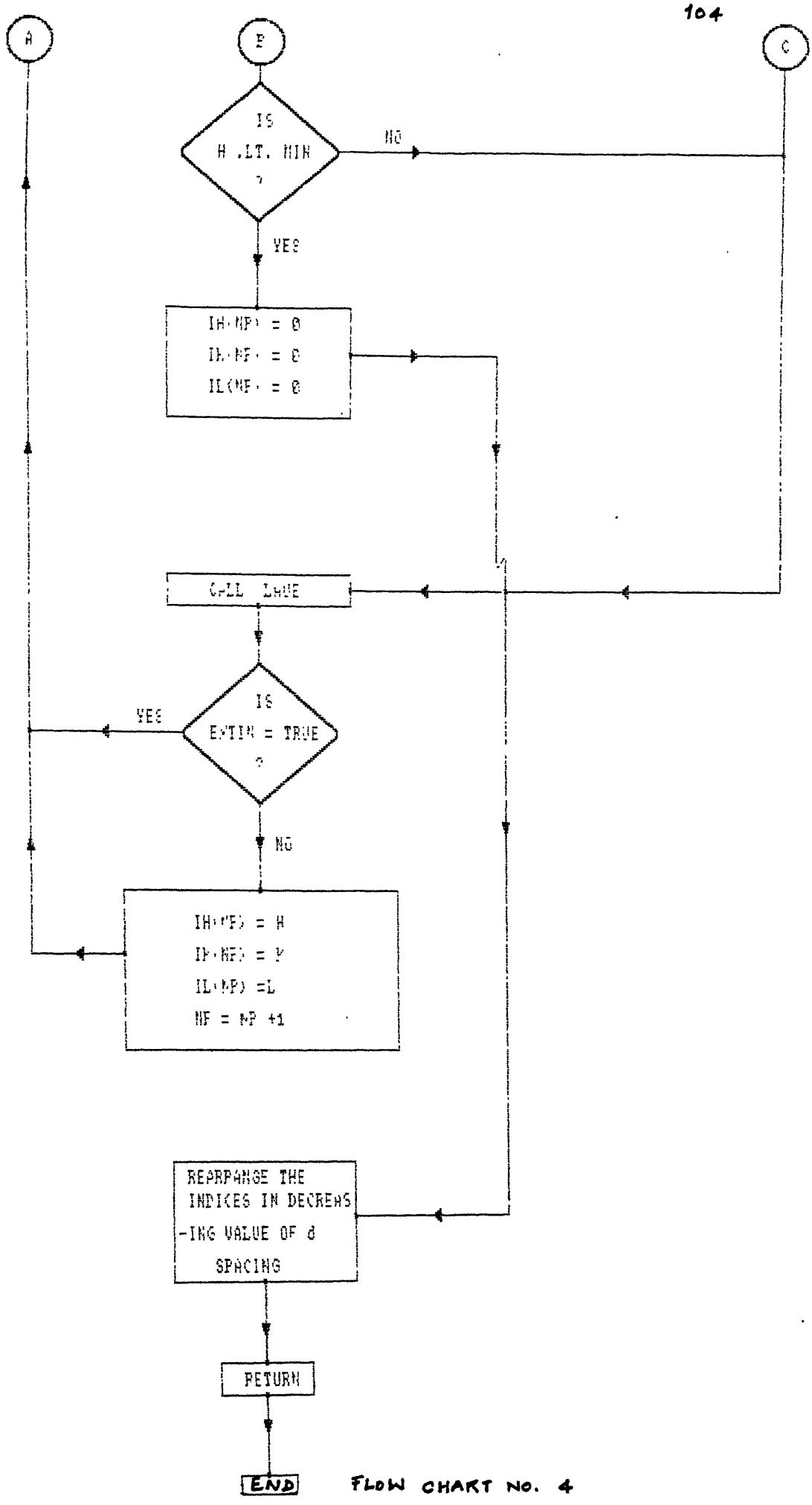
SUBROUTINE ZANGL(MZN,IZ)



FLOW CHART NO. 3

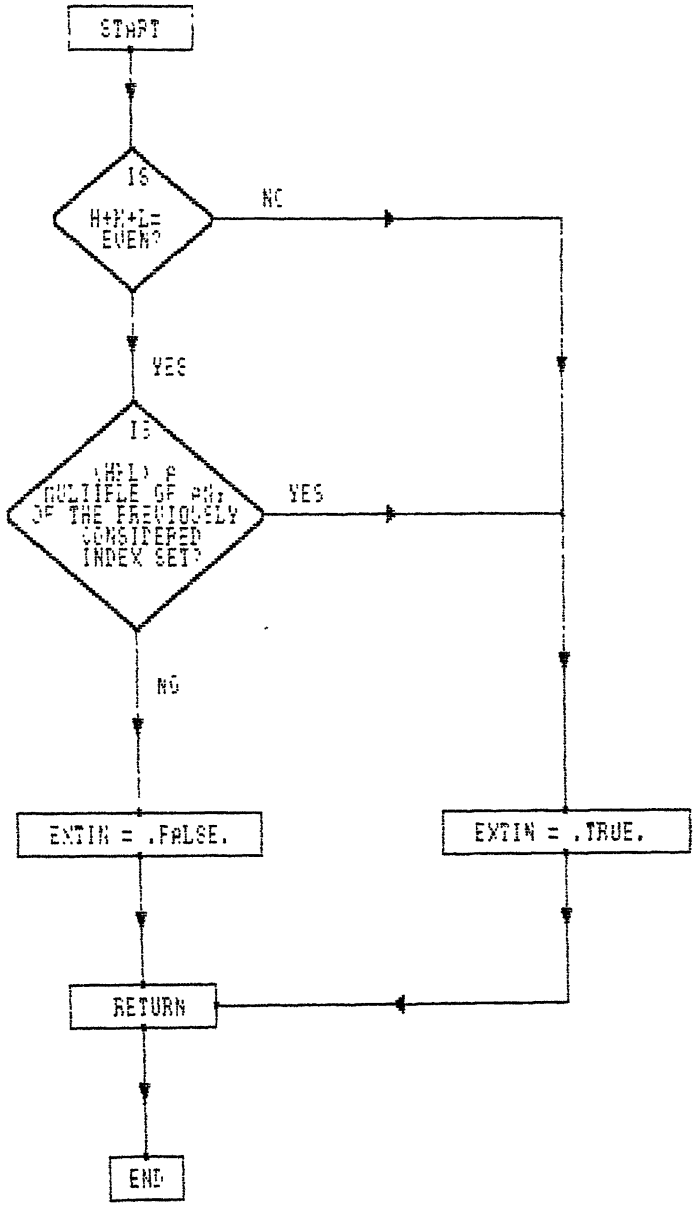
SUBROUTINE SELECT



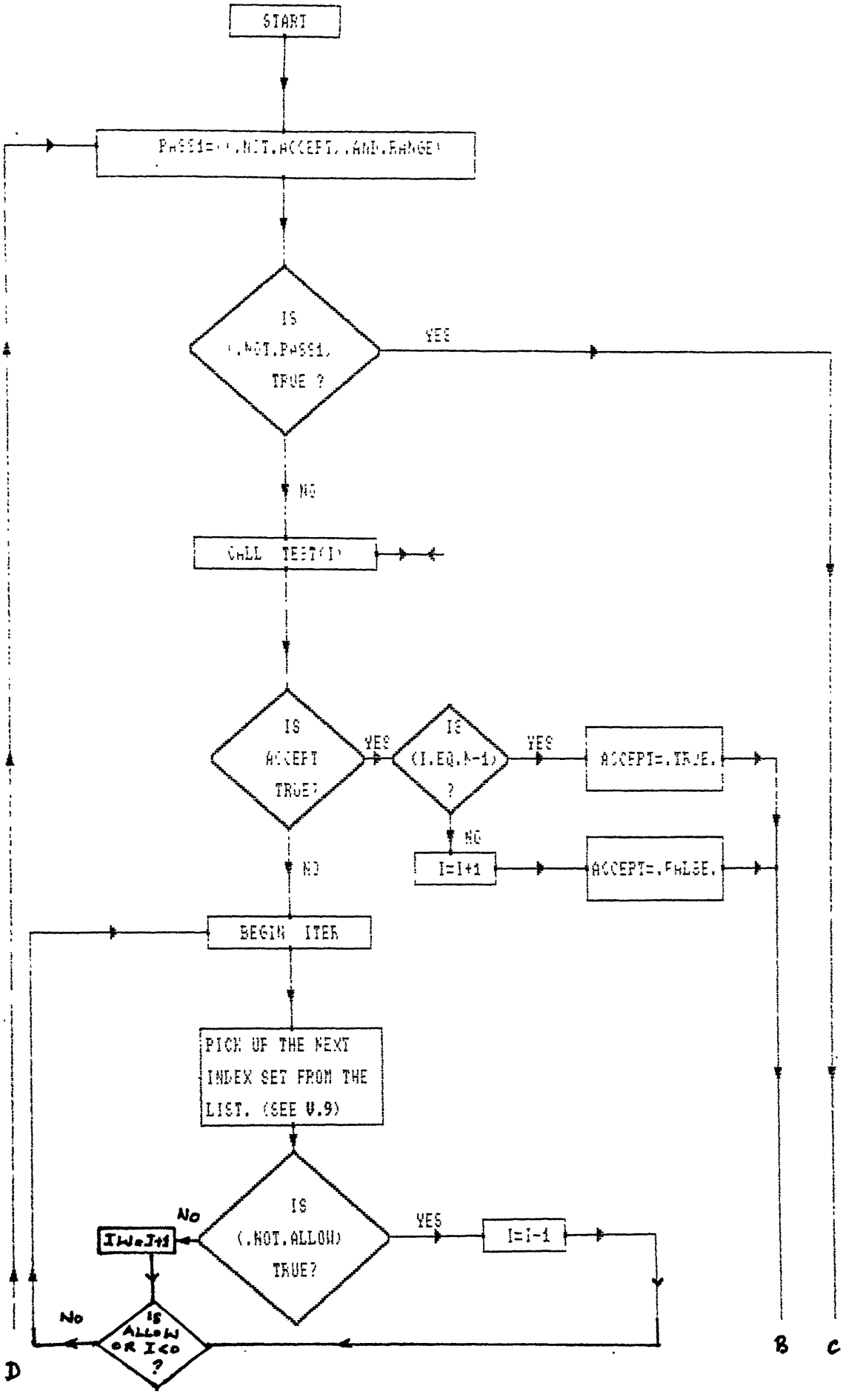


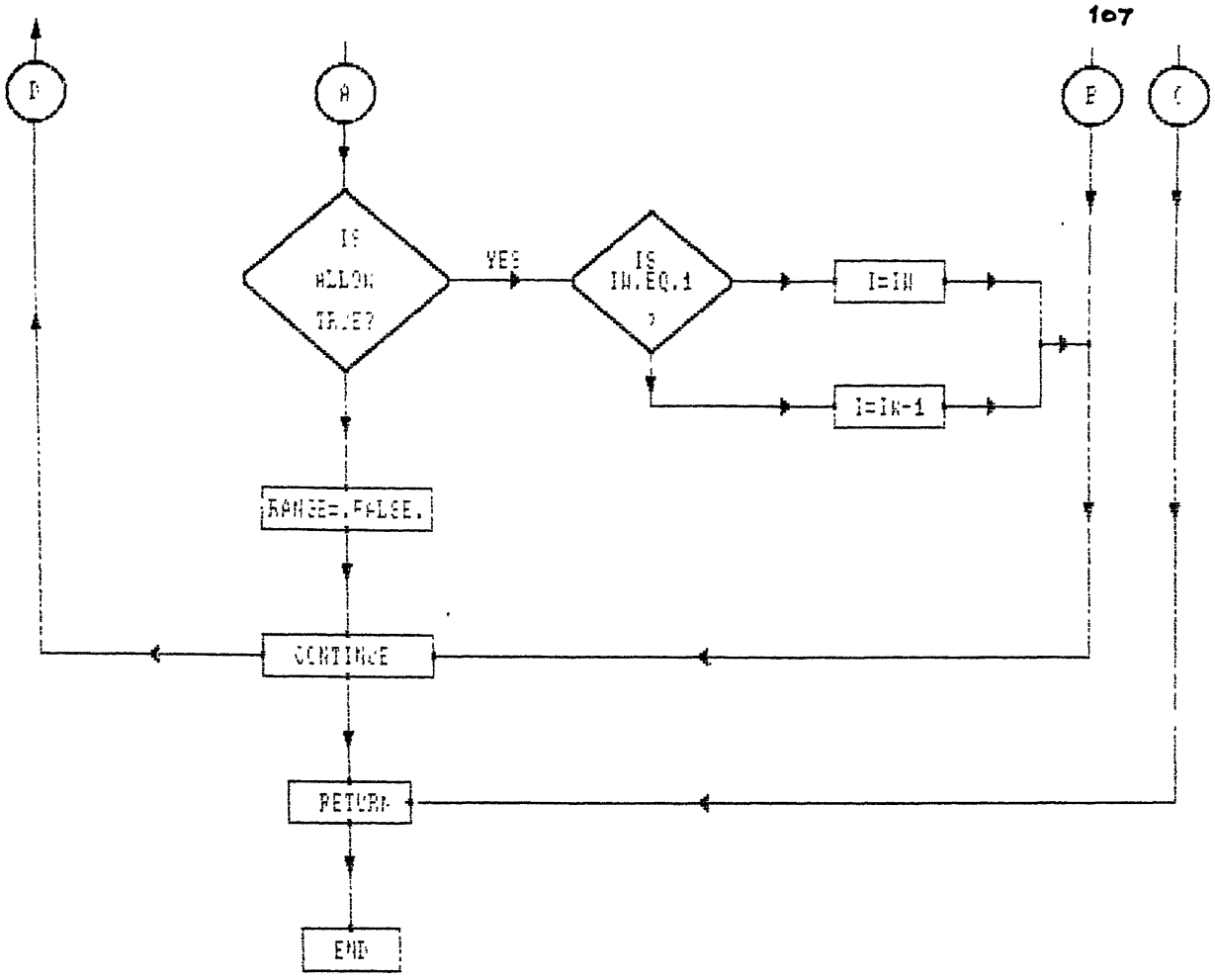
FLOW CHART NO. 4

SUBROUTINE LAUE

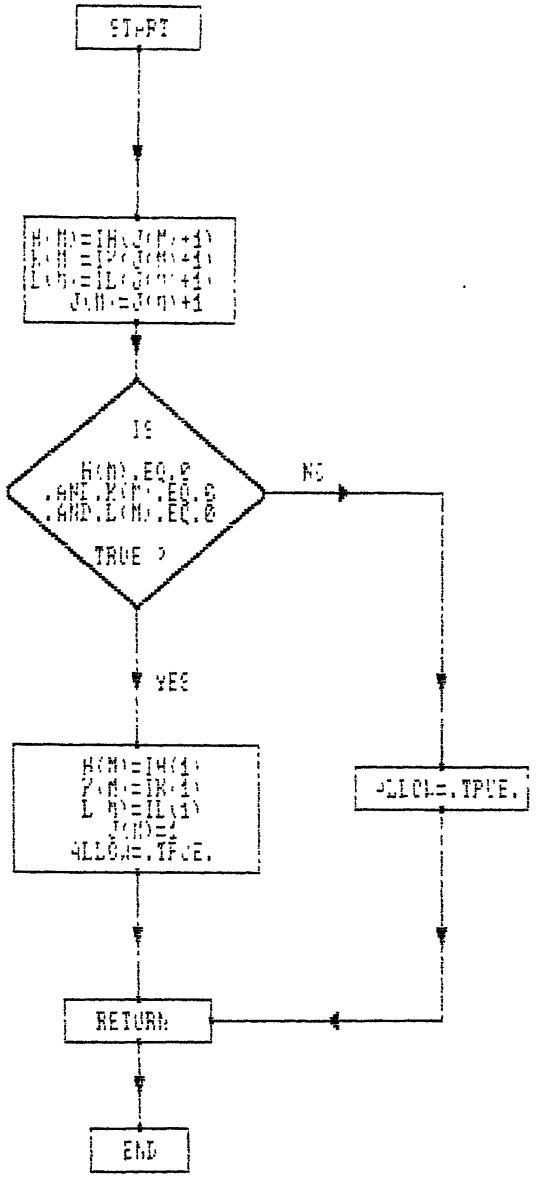


FLOW CHART NO. 5


START
PASS1=(.NOT.ACCEPT.AND.RANGE)
IS (.NOT.PASS1. TRUE ?
YES
NO
CALL TEST(I)
IS ACCEPT TRUE?
YES
IS (I.EQ.N-1) ?
YES
ACCEPT=.TRUE.
NO
I=I+1
ACCEPT=.FALSE.
NO
BEGIN ITER
PICK UP THE NEXT INDEX SET FROM THE LIST. (SEE U.9)
IS (.NOT.ALLOW) TRUE?
YES
I=I-1
No
IW=J+1
IS ALLOW OR I<0 ?
No
B
C
D

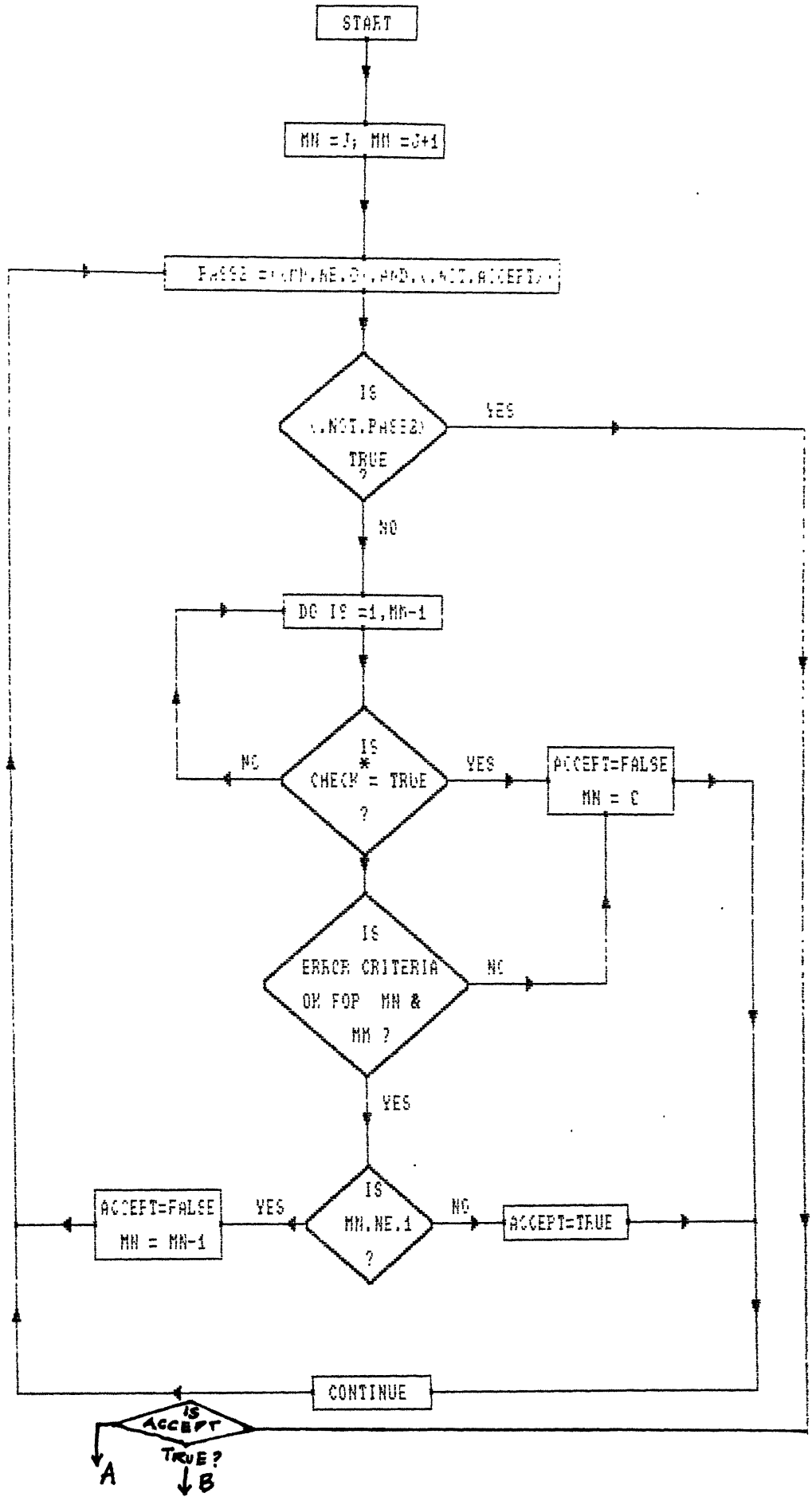


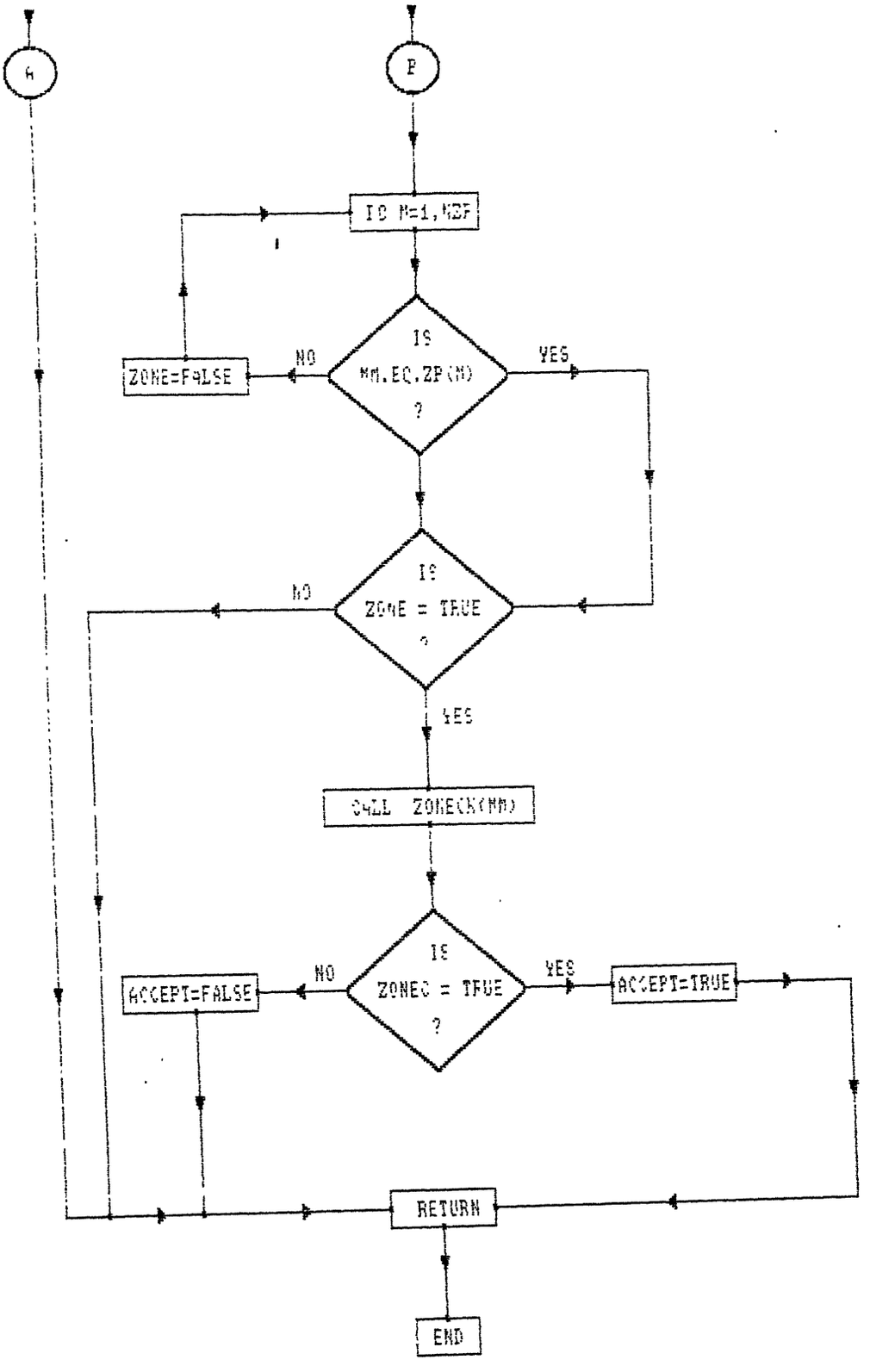
FLOW CHART NO. 6.



FLOW CHART FOR PICKING THE
NEXT FROM THE LIST
(USED BY SUBROUTINE INDEX)

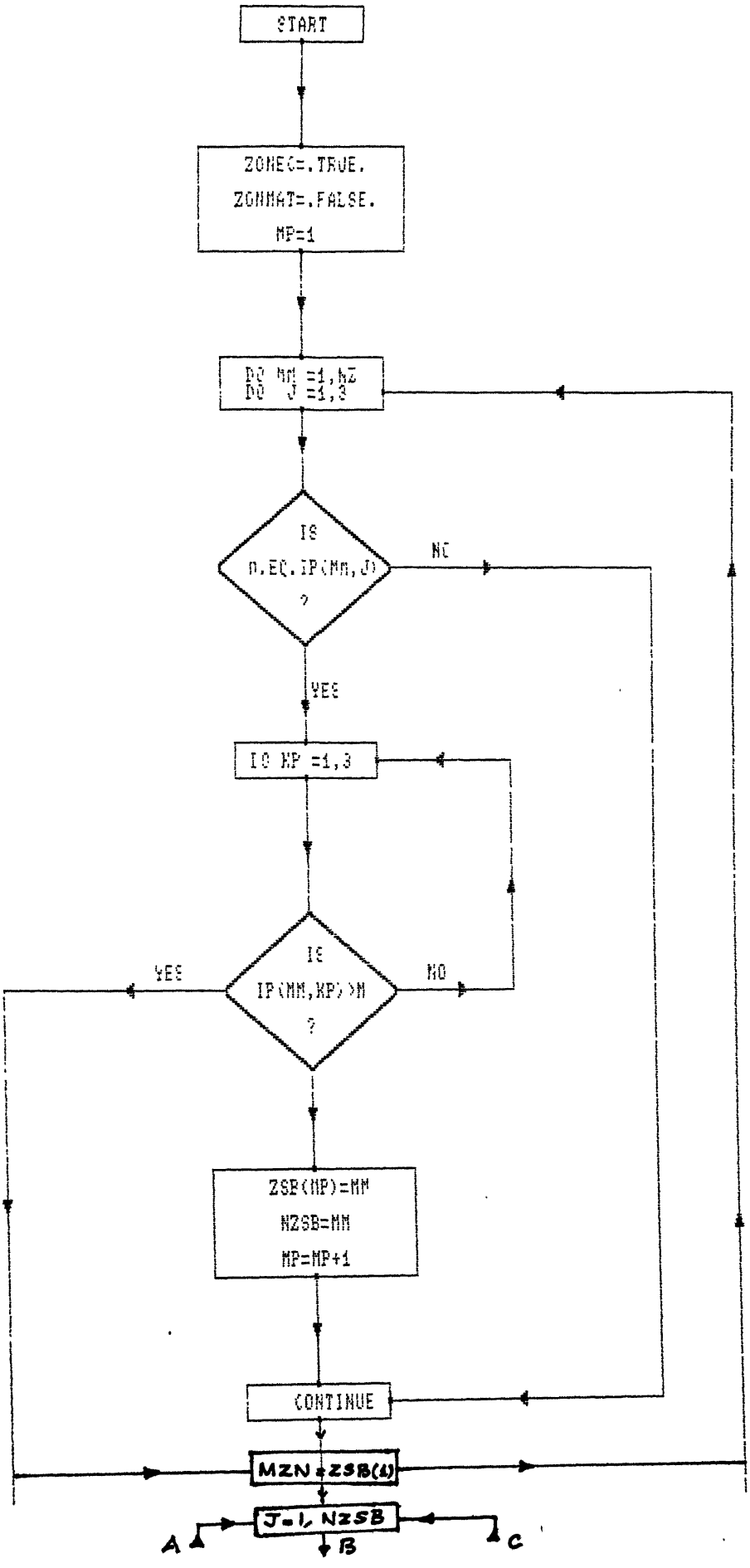
SUBROUTINE TEST(J)

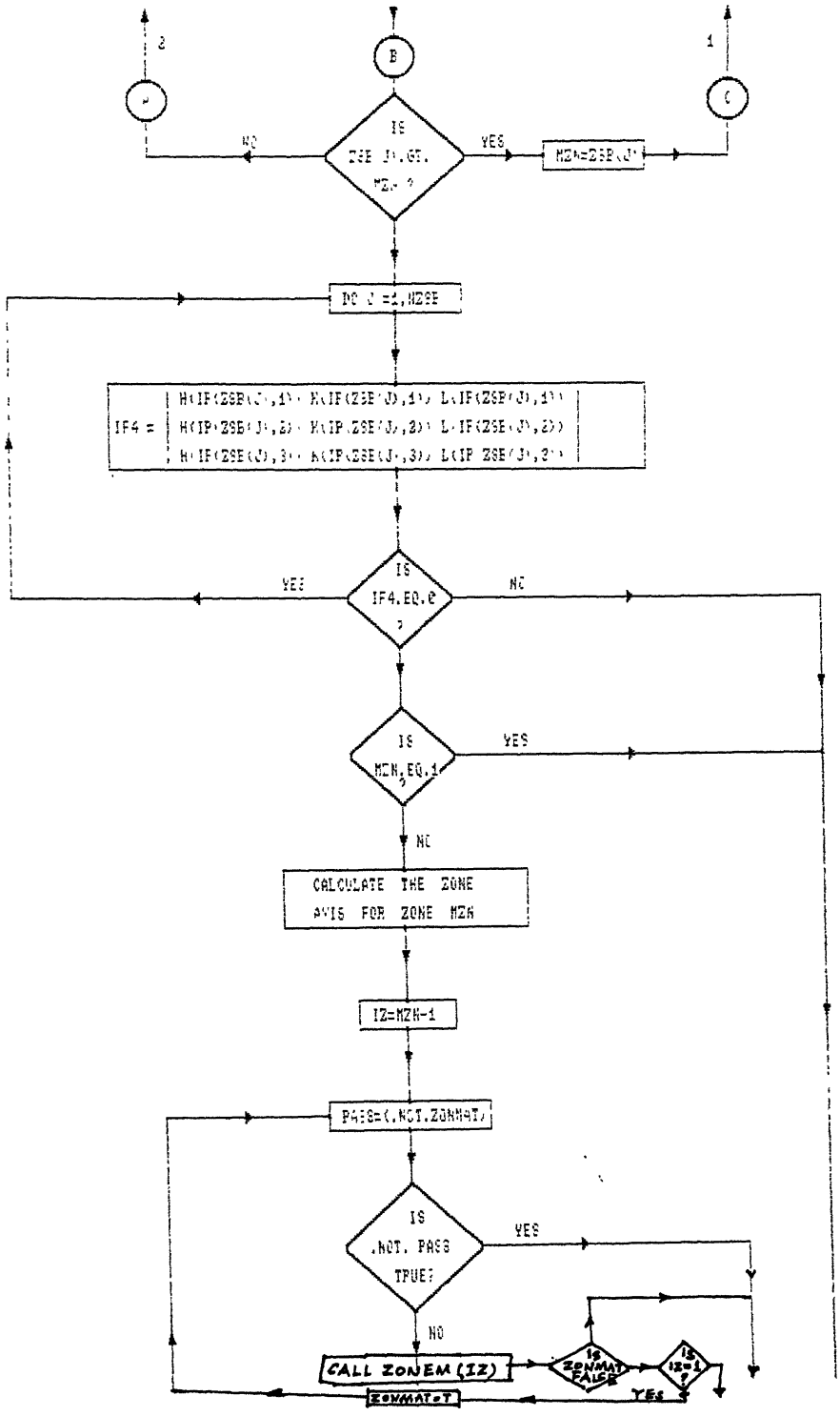


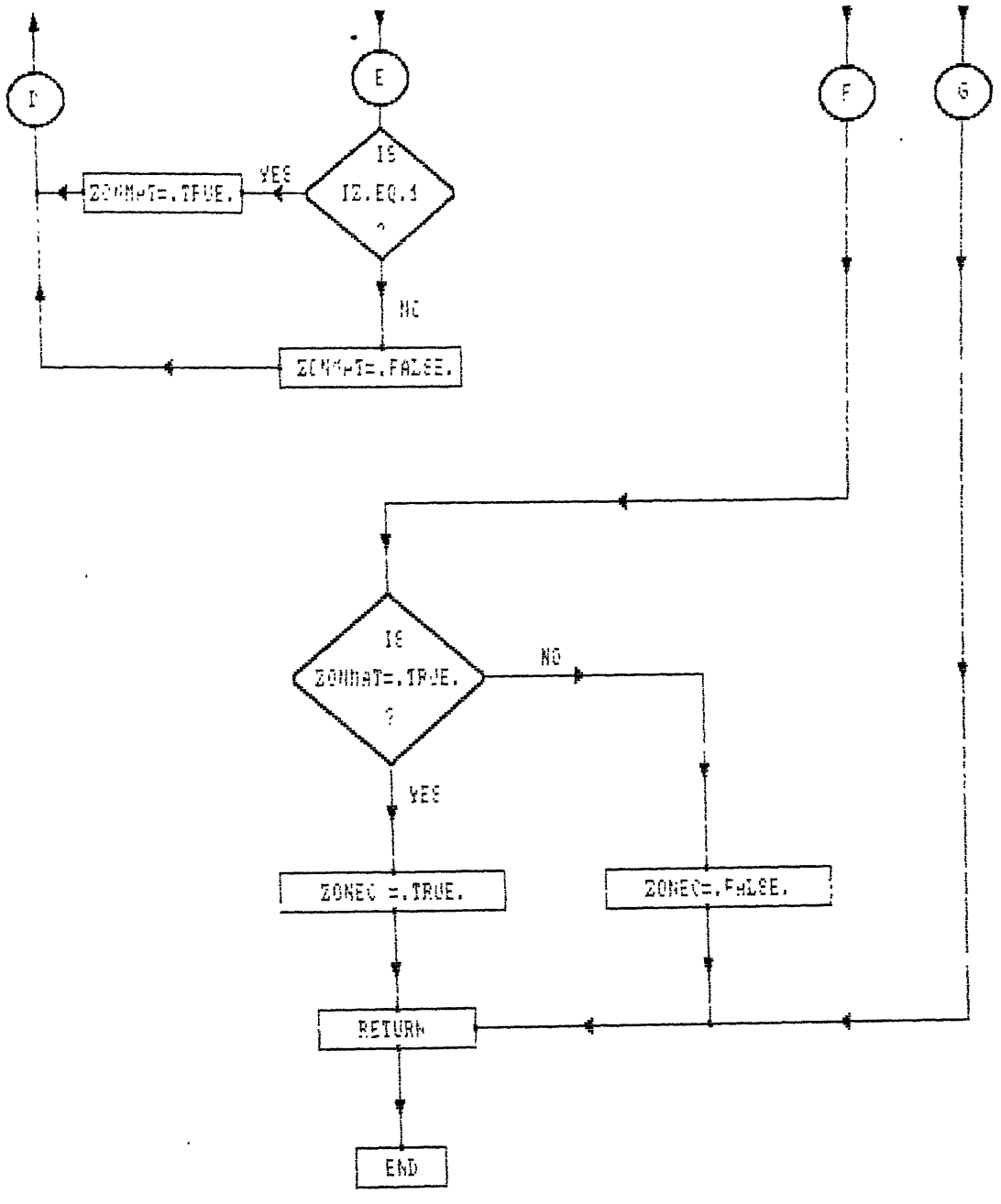


FLOW CAHRT NO. 7.

SUBROUTINE ZONECK(M)

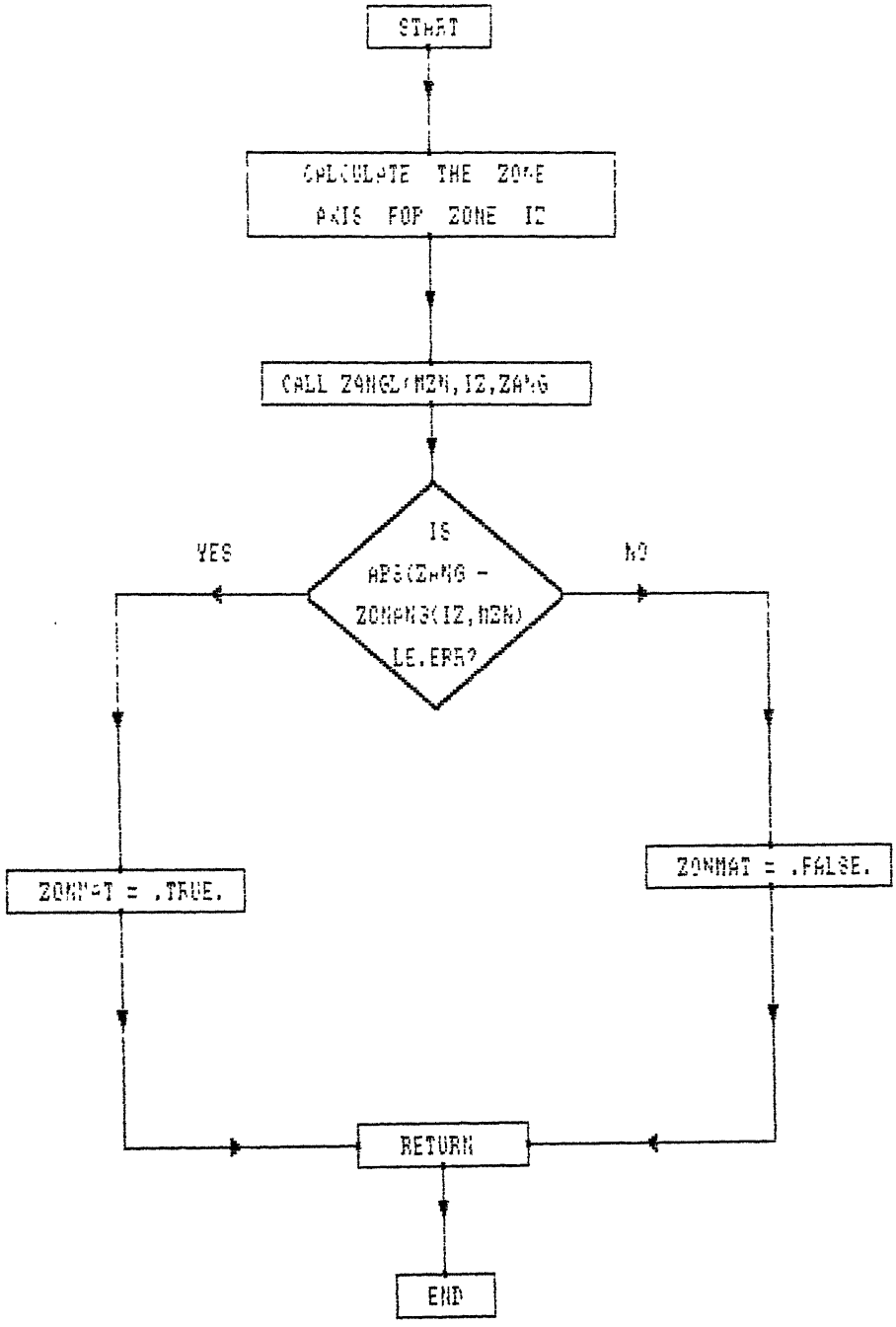



2
B
1
A
C
IS
ZSE J>.GT.
MZN ?
NO
YES
MZN=ZSE(J)
DO J =1,NZSE
IF4 =
H(IF(ZSE(J),1)) M(IF(ZSE(J),1)) L(IF(ZSE(J),1))
H(IF(ZSE(J),2)) M(IF(ZSE(J),2)) L(IF(ZSE(J),2))
H(IF(ZSE(J),3)) M(IF(ZSE(J),3)) L(IF ZSE(J),3))
IS
IF4.EQ.0
?
YES
NO
IS
MZN.EQ.1
YES
NO
CALCULATE THE ZONE
AXIS FOR ZONE MZN
IZ=MZN-1
PASS=(.NOT.ZONMAT)
IS
.NOT. PASS
TRUE?
YES
NO
CALL ZONEM (IZ)
IS
ZONMAT
FALSE
IS
IZ=1
?
YES
ZONMAT=T



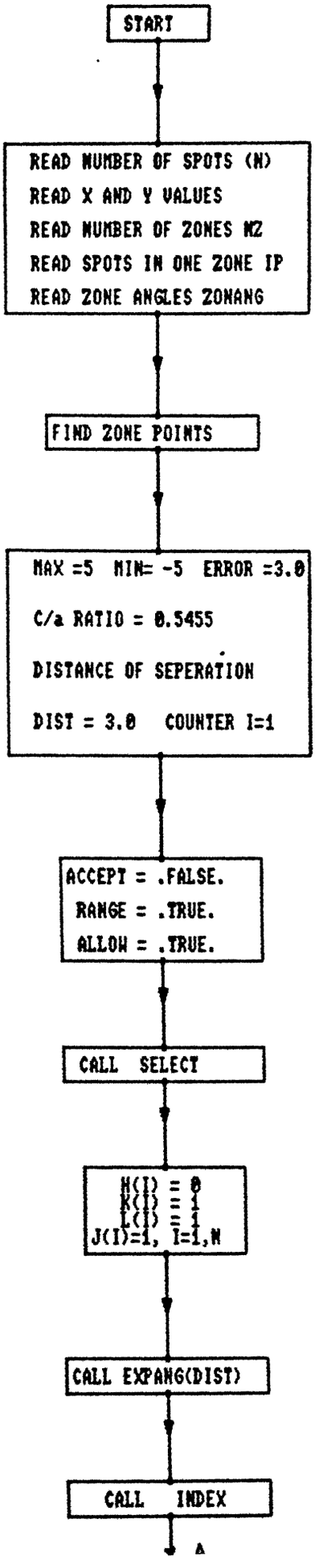
FLOW CHART NO. 8

SUBROUTINE ZONEM(IZ)



FLOW CHART NO. 9

MAIN PROGRAM



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